EVALUATION OF BITUMINOUS MATERIALS USED IN PAVEMENT RECYCLING PROJECTS AT TYNDALL, MACDILL, AND HURLBURT AIR FORCE BASES

B. KIGGUNDU, R. MARTINEZ, B. HUMPHREY, T. SHULER

NEW MEXICO ENGINEERING
RESEARCH INSTITUTE
BOX 25, UNIVERSITY OF NEW MEXICO
ALBUQUERQUE NM 87131

JULY 1987

FINAL REPORT

FEBRUARY 1985 - DECEMBER 1986

DTIC
ELECTE
NOV 1 6 1987
S
E
D

AFESC

**ENGINEERING & SERVICES LABORATORY
AIR FORCE ENGINEERING & SERVICES CENTER
TYNDALL AIR FORCE BASE, FLORIDA 32403**

87 10 27 065

REPORT DOCUMENTATION PAGE

A188 068

1a. REPORT SECURITY CLASSIFICATION	1b. RESTRICTIVE MARKINGS
Unclassified	

2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT
	Approved for public release:
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	Distribution is unlimited.

4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S)
NMERI WA5-11(5.07)	ESL-TR-86-50

6a. NAME OF PERFORMING ORGANIZATION	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION
New Mexico Engineering Research Institute	NMERI	Engineering and Services Laboratory

6c. ADDRESS (City, State and ZIP Code)	7b. ADDRESS (City, State and ZIP Code)
Box 25, University of New Mexico Albuquerque, New Mexico 87131	Air Force Engineering and Services Center Tyndall Air Force Base, Florida 32403

8a. NAME OF FUNDING/SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
HQ AFESC	RDCP	Contract No. F29601-84-C-0080

8c. ADDRESS (City, State and ZIP Code)	10. SOURCE OF FUNDING NOS.			
	PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT NO.
Tyndall AFB FL 32403-6001	63723F	2104	1A	29

11. TITLE (Include Security Classification)
EVALUATION OF BITUMINOUS MATERIALS USED IN PAVEMENT RECYCLING PROJECTS AT TYNDALL, MACDILL,

12. PERSONAL AUTHOR(S)
B. Kiggundu, R. Martinez, B. Humphrey, and T. Shuler

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Yr., Mo., Day)	15. PAGE COUNT
Final Report	FROM Feb 85 TO Dec 86	July 1987	200

16. SUPPLEMENTARY NOTATION
Availability of this report is specified on reverse of front cover.

17.	COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB. GR.	Flexible pavements, reclaimed asphalt pavement (RAP), open graded friction course (OGFC), hot recycling, aged binders, modifiers, compatibility, modifier selection
13	02		
11	08		

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This report presents results of a study involving bituminous materials from Tyndall and MacDill Air Force Bases and Hurlburt Field. These materials included Reclaimed Asphalt Pavement (RAP), modifiers, virgin asphalts, and new aggregates. A tentative modifier selection criterion was used to judge the quality of materials used in the recycling efforts at the respective sites.

The results showed that independent adequacy of physical properties from chemical properties in selection of modifiers could not be established. However, some of the results showed that physical properties were more sensitive indicators of changes in binders due to aging.

In addition, this report includes the tentative modifier selection criteria and results of an interlaboratory study from which variability limits to parameters determined using modified Clay-Gel and Heithaus procedures are established. The modified Clay-Gel and Heithaus procedures are included.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT	21 ABSTRACT SECURITY CLASSIFICATION
UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS ☐	Unclassified

22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE NUMBER (Include Area Code)	22c. OFFICE SYMBOL
Capt Martin D Lewis	(904) 283-6317	HQ AFESC/RDCP

DD FORM 1473, 83 APR EDITION OF 1 JAN 73 IS OBSOLETE.

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE

11. (Title Continued)

AND HURLBURT AIR FORCE BASES

18. (Subject Terms Continued)

specification, chemical/physical properties, Clay-Gel modified test, Heithaus
solubility test, variability limits, pilot study, viscosity temperature susceptibility,
asphaltenes plus saturates, asphaltene state of peptization, viscosity aging index,
recycled pavement performance, recycled asphalt concrete (RAC), Marshall properties,
resilient moduli.

Accession For	
NTIS GRA&I	☒
DTIC TAB	☐
Unannounced	☐
Justification	
By	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
A-1	

PREFACE

This final report was prepared by the New Mexico Engineering Research Institute, University of New Mexico, at the Eric H. Wang Civil Engineering Research Facility, Kirtland Air Force Base, New Mexico under Contract F29601-84-C-0080, Subtasks 5.05 and 5.07, for the Pavements Technology Branch, Engineering and Services Laboratory, Headquarters Air Force Engineering and Services Center, Tyndall Air Force Base, Florida.
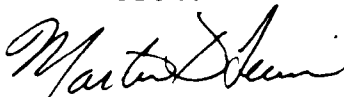
This report summarizes the work done between February 14, 1985 and December 31, 1986. Mr James G. Murfee and Capt Martin D. Lewis were the HQ AFESC/RDCP project officers.

The authors wish to thank Capt R. F. Wade of MacDill AFB, Capt M. Wolfe of Tyndall AFB, and Mr R. Stephens of Hurlburt Field for assistance with project materials; Mr S. C. Mawby for discussions about plant and construction operations; Mr J. C. Darnell for discussions regarding paving operations and provision of quality control data from Hurlburt Field; Dr R. D. Pavlovich and Mr R. G. McKeen for discussions and helpful suggestions; Dr R. D. Pavlovich for formalizing the procedures in Appendix B; and T. Escobedo, G. Kiscaden, D. Gonzales, B. Wiuff, S. Scales, and F. Heckes for assistance with material evaluation.
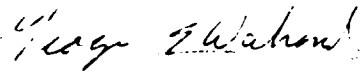
The authors thank the following organizations: Petroleum Sciences, Inc; Chicago Testing Laboratory; and Waterways Experiment Station for participating with the New Mexico Engineering Research Institute in testing various bituminous materials. These organizations generated data to establish variability limits for the methods listed in Appendix B. The results of their efforts, the analyses, and the proposed variability limits are contained in Appendix A.

This report has been reviewed by the Public Affairs Office (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication.

MARTIN D. LEWIS, Capt, USAF
Project Officer

ROBERT R. COSTIGAN, Lt Col, USAF
Chief, Engineering Research
  Division

GEORGE E. WALROND, Maj, USAF
Chief, Pavement Technology
  Branch

LAWRENCE D. HOKANSON, Col, USAF
Director, Engineering and Services
  Laboratory

iii
(Reverse of this page is blank)

TABLE OF CONTENTS

TABLE OF CONTENTS (CONTINUED)

TABLE OF CONTENTS (CONCLUDED)

LIST OF FIGURES

LIST OF TABLES

LIST OF TABLES
(CONCLUDED)

SECTION I
INTRODUCTION

A. OBJECTIVES

The objective of this research is to evaluate asphalt modifiers used in recycling projects at Tyndall and MacDill Air Force Bases and Hurlburt Field. The intent was to judge the adequacy of certain physical test results and compare them to the adequacy of chemical components of modifiers to predict performance attributes of the recycled pavement. The tentative modifier selection criteria used in the judgement and variability limits proposed from an interlaboratory pilot study are included in this report as Appendix A.

B. BACKGROUND

Research indicates that recycled pavements may have less tendency to crack at low temperatures than virgin mixes (Reference 1). Other research indicates that recycled mixes harden at a slower rate than control mixes when measured by resilient modulus (Reference 2). Although no field data have been collected to verify laboratory findings, the indication is that recycled mixes may possess certain superior properties over virgin mixes.

Achievement of these improved properties seems to depend on several factors:

- Evaluation of Reclaimed Asphalt Pavement (RAP)
- Selection of modifiers
- Selection of virgin asphalt
- Selection of virgin aggregate

The modifier has the greatest influence on binder characteristics, particularly rheology. Under similar service conditions modifiers which produce the same initial binder viscosity can affect pavement performance quite differently during service (Reference 3) and in binder performance in the laboratory (Reference 4).

1

Data presented in this report are based on tentative recycling agent selection criteria (Reference 4) used to evaluate recycled asphalt-concrete from MacDill and Tyndall Air Force Bases and Hurlburt Field. Modifications to the tentative recycling agent selection criteria and results of variability limits development efforts are included in this report.

The Tyndall recycled concrete materials were produced only in the laboratory because the Air Force chose to place a conventional mix and sold the RAP millings to the contractor. The modifiers used at MacDill Air Force Base and Hurlburt Field were not preselected in accordance with the tentative modifier selection criteria. The modifiers for the Tyndall Air Force Base project had been selected to test the criteria.

C. SCOPE

This research was performed under Contract No. F29601-84-C-0080, Subtask Statement 5.07. Various materials were evaluated in conjunction with the objective of the research as shown below:

- RAP
- Asphalt recycling agents (modifiers)
- Virgin asphalt and aggregate
- Reclaimed asphalt
- Reclaimed modified asphalt
- Recycled Asphalt Concrete (RAC)

This report evaluates recycled pavement materials from Tyndall and MacDill Air Force Bases and Hurlburt Field. The effort was conducted by the New Mexico Engineering Research Institute (NMERI), Albuquerque, New Mexico.

SECTION II
MATERIALS

Materials evaluated by this research were obtained from Tyndall and MacDill Air Force Bases and Hurlburt Field. The types of materials evaluated are outlined below, although not all materials listed may have been evaluated at each base:

- Drilled cores obtained prior to recycling
- Cold-milled RAP
- RAP modifiers
    AC-10
    AC-20
    Recycling agents
- Virgin asphalts
- Virgin aggregates
- RAC

The distinction between RAP and RAC should be clarified. RAP is the asphalt concrete immediately after milling, before modifiers are added. RAC is the RAP after modifiers and virgin aggregates have been added. Use of the term RAC does not necessarily infer the material has been placed as a pavement.

A description of the materials obtained from each Air Force Base (AFB) and Hurlburt Field follows.

A. TYNDALL AFB

1. Cores

Thirty 4-inch-diameter cores were supplied to NMERI from Runway 13R-31L by the Air Force Engineering and Services Center (AFESC) project officer for preliminary analysis. The 30 cores were even-numbered. These cores were obtained at 385-foot intervals along the runway as represented in Figure 1. Certain cores were selected from trafficked and nontrafficked runway locations for special testing described later. These cores are shaded on Figure 1.

3
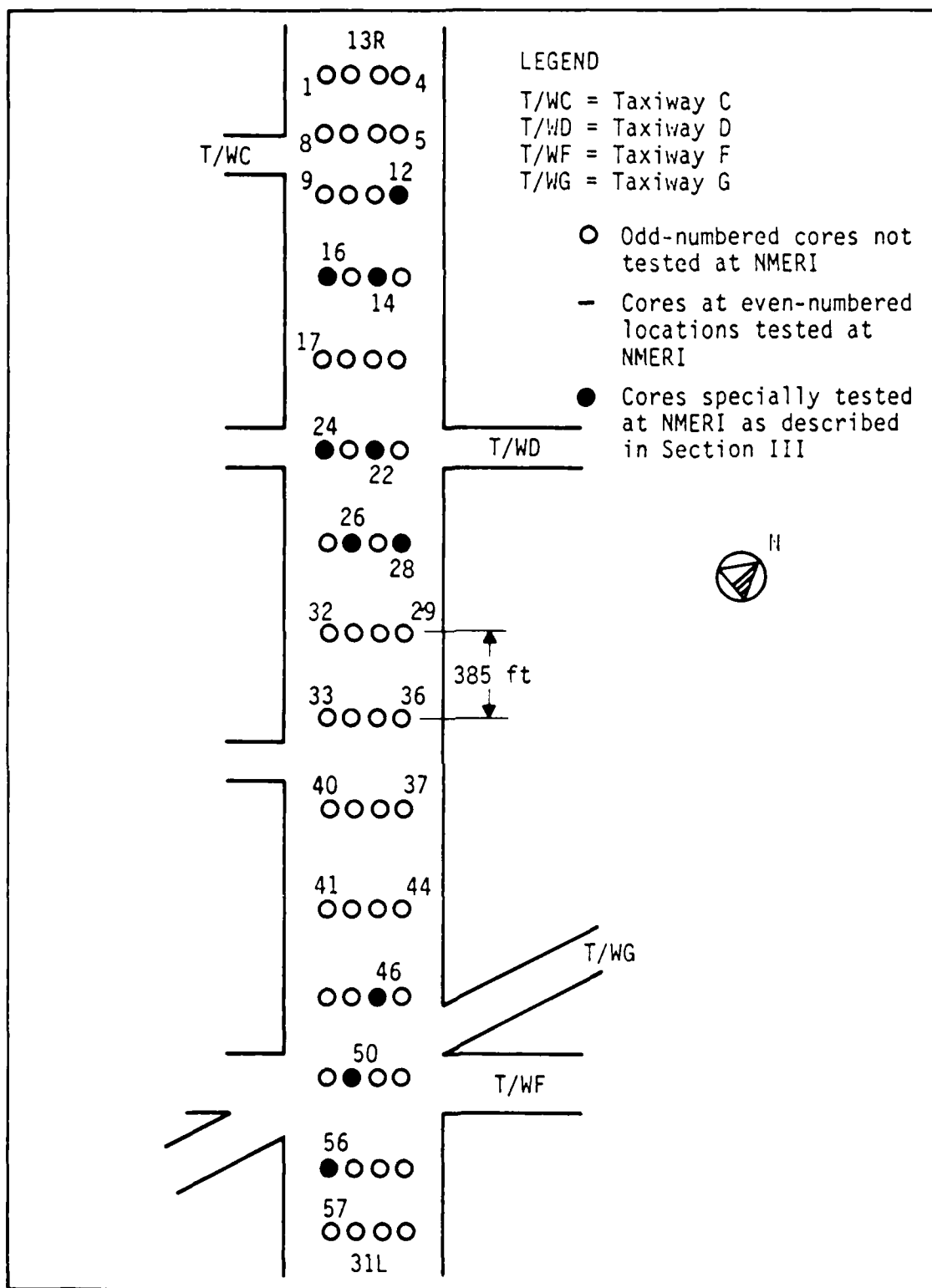
Figure 1. Tyndall AFB Core Locations.

4

Runway 13-31 consisted of an asphalt-concrete pavement and open-graded friction course (OGFC) prior to rehabilitation. Cold milling operations removed various thicknesses of the old surface, including both OGFC and dense-graded material, to produce a level base for overlay construction. This cold-milled material is referred to as RAP. Because of time constraints no RAP was evaluated for the Tyndall project.

The surface of the pavement consisted of 0.5 to 1.0 inches of OGFC while the underlying course was dense-graded asphalt concrete. Since the field RAP included both OGFC and dense-graded materials, both types of materials were mixed in the laboratory before further evaluation.

2. Modifiers

Three 1-gallon samples of modifiers were obtained by NMERI through the AFESC project officer. Modifiers consisted of three recycling agents. The recycling agents submitted for evaluation were petroleum-based products selected to reflect a broad range of such materials used in western Florida. Recycling agents are referred to as MBD-A, MBD-B, and MBD-C.

3. Virgin Asphalt

An AC-20 asphalt was supplied to NMERI for evaluation by the AFESC. This asphalt was to be used in recycled mixtures in case additional virgin binder was required due to use of virgin aggregate.

4. Virgin Aggregates

Virgin mineral aggregates were added to the RAP in proportions of 30:70, 40:60, and 50:50 for RAP and virgin aggregates, respectively. Aggregates were described as Alabama limestone.

B. MACDILL AFB

1. Introduction

Two construction phases occurred during work at MacDill. Phase A refers to reconstruction of Taxiways 3, 4, 7, 9, 10, 11, 12, 16, and the

5

junction Taxiways 5 and 36. Phase B refers to work on the connecting taxiway
between Taxiways 7 and 36, Taxiways 4 to 5, between Taxiway 5 and the south
apron, north and south aprons, and part of the junction between Taxiways 5 and
36.

2. Cores

Fourteen 6-inch-diameter cores from Taxiway 7 were supplied to NMERI
by the base personnel. These cores were obtained from six locations as shown
in Figure 2.

All cores were obtained from the Taxiway 7 pavement after recycling
operations were completed in Phase A.

3. Milled RAP

All RAPs studied were supplied to NMERI by base personnel from Phase
B of the reconstruction effort at MacDill. Two types of RAP were supplied.
"In-use RAP" refers to material recycled and replaced in the pavement.
"Rejected RAP" refers to RAP not recycled for further use. Aggregate in the
rejected RAP consisted of gray slag. This material was identified during
Phase A reconstruction as a source of excessive fine material in the original
pavement.

4. Modifiers

AC-10 and AC-20 virgin asphalts, identified here as MBD-D and MBD-E
and two recycling agents, MBD-F and MBD-G, respectively, were supplied to
NMERI by base personnel. All four modifiers were used during Phase A
reconstruction. MBD-D was the only modifier used during Phase B
reconstruction.

Locations on Taxiway 7 where the four modifiers were used were not
supplied to NMERI.

Notes: Numbers 7-1 through 7-6 are approximate core locations along Taxiway 7.

Numbers 3 through 36 indicate taxiways.
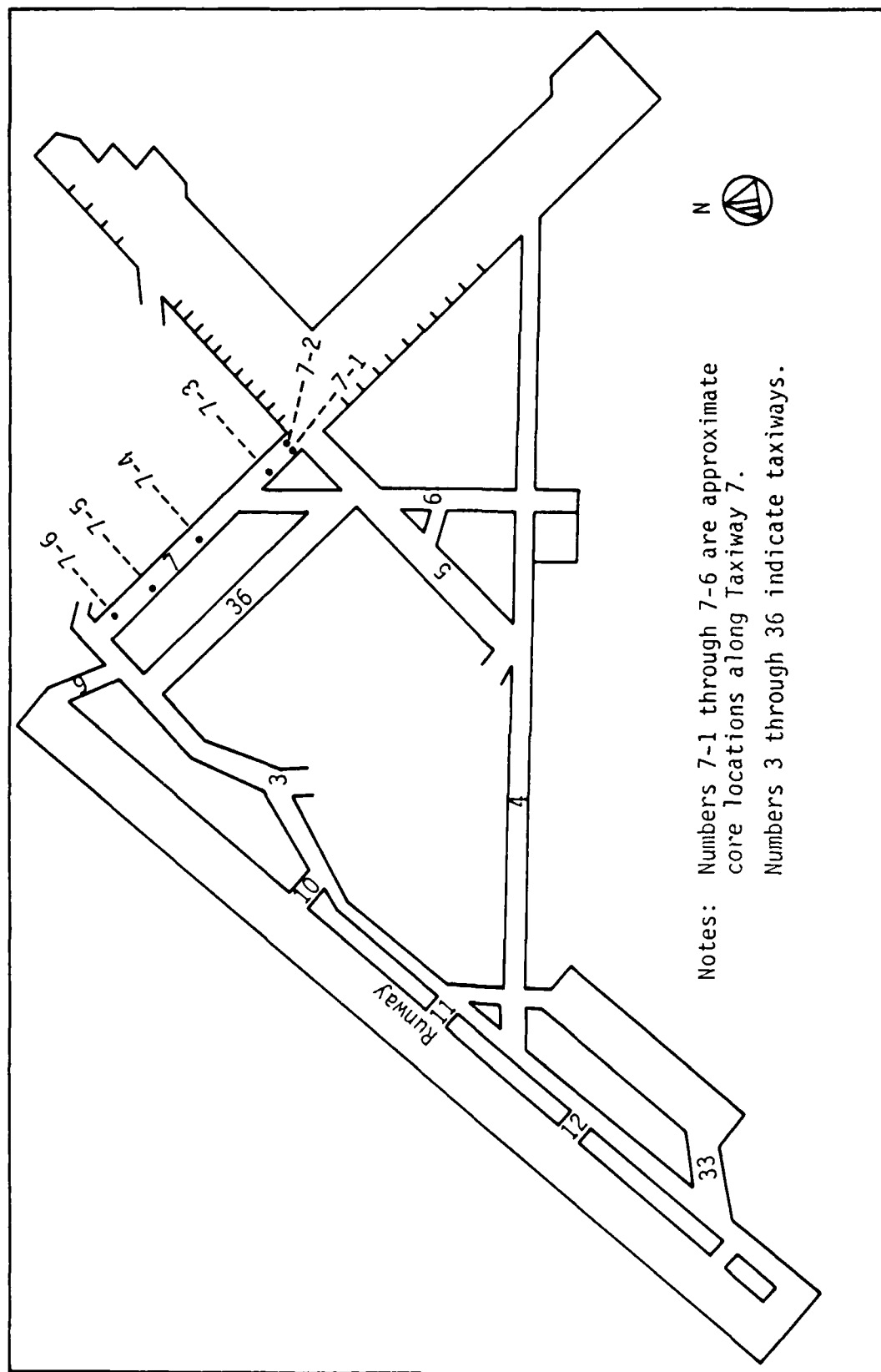
Figure 2. MacDill AFB Site Plan.

C.   HURLBURT FIELD

1.   Milled RAP

The north-south runway was cold-milled between the months of May to
June 1985 and the milled RAP stockpiled. Samples of the stockpile were
obtained for testing by the project officer.

2.   Modifiers

Two modifiers were supplied to NMERI through the project officer. The
first, Chevron AC 2.5, is designated MBD-2D, and the second, Chevron AC 2.5
containing 0.5 percent antistripping agent, is designated MBD-2DD. Type of
liquid antistripping agent is unknown. The paving contractor reported the
modifiers were from the same source. Full-scale recycling at Hurlburt report-
edly used MBD-2DD.

3.   Loose RAC

Loose recycled asphalt-concrete was obtained from a transport truck after
loading at the hot-mix plant. Samples were taken from each of the four quad-
rants of the truck bed. RAC in this truck was placed approximately in loca-
tion TM shown in Figure 3.

4.   RAC Cores

Cores of the RAC pavement were obtained by the paving contractor and
supplied to NMERI by Hurlburt personnel. Seven 4-inch-diameter cores were
supplied to NMERI by base personnel and tests conducted at NMERI in New
Mexico. Seventeen additional cores were obtained and tested by the paving
contractor. Core locations are unknown.

5.   Virgin Aggregates

Virgin aggregates added to RAP during recycling are described as S-1
stone, stone screenings, and washed sand.

Figure 3. Hurlburt Field Runway Site Plan.
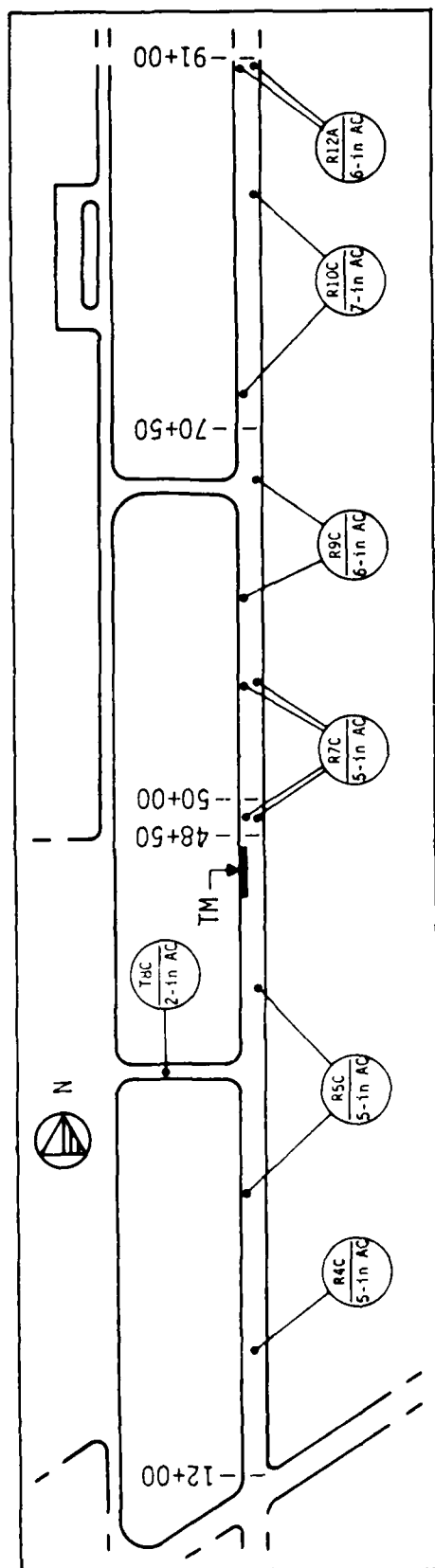
R4C, 5-in AC — Runway, keel interiors; 5 inches asphalt-concrete (AC) pavement

R7C, 5-in AC — Runway, edges interior; 5 inches AC pavement

R10C, 7-in AC — Runway, edges interior, 7 inches AC pavement

T8C, 2-in AC — Taxiway C; 2 inches AC pavement

R5C, 5-in AC — Runway, edges interiors; 5 inches AC pavement

R9C, 6-in AC — Runway, edges interior; 6 inches AC pavement

R12A, 6-in AC — Runway edges, north end; 6 inches AC pavement

TM — Approximent location of truck, sampled

SECTION III
MATERIALS TESTING

A. SUMMARY

An extensive number of tests and types of materials were evaluated in accordance with the scope of this project. Therefore, a summary of the tests performed for each type of material is included below. Certain materials were evaluated or obtained for special purposes; therfore, a description is included when necessary to clarify various aspects of this section.

B. TYNDALL AFB

1. Cores

- Bulk and apparent density   ASTM D 2726   (Reference 11)
- Binder extraction   ASTM D 2172   (Reference 11)
- Binder recovery   ASTM D 1856 modified (Reference 5)

2. Core Binder

- Penetration   ASTM D 5   (Reference 17)
- Viscosity   ASTM D 2171   (Reference 11)
- Composition   ASTM D 2007 modified (References 4,12)
- Solubility   Heithaus modified (References 4,6)

3. Core Aggregates

- Gradation   ASTM C 136   (Reference 11)
- Specific Gravity   ASTM C 127 and 128   (Reference 11)

4 Modifiers (Virgin AC-20 and Recycling Agents)

- Viscosity   ASTM D 2170 and 2171 (Reference 11)
- Composition   ASTM D 2007 modified (References 4,12)
- Solubility   Heithaus modified (References 4,6)

5. Modifiers (Recycling Agents Only)

- Weight loss   ASTM D 2872   (Reference 11)
- Flash Point   ASTM D 92   (Reference 11)

6. Modifier/Core Binder Blends (Before RTFO)

- Viscosity   ASTM D 2170, D 2171, D 3381
                              (Reference 11)
- Penetration   ASTM D 5   (Reference 11)
- Rheology   Schweyer apparatus (Reference 7)
- Ductility   ASTM D 113   (Reference 11)
- Composition   ASTM D 2007 modified (References 4,12)
- Solubility   Heithaus modified (References 4,6)

7. Modifier/Core Binder Blends (After RTFO)

- Penetration   ASTM D 5   (Reference 12)
- Ductility   ASTM D 113   (Reference 12)
- Viscosity   ASTM D 2171   (Reference 12)
- Composition   (References 4, 12)
- Solubility   (References 4, 6)

8. RAC

- Mix design   Tentative recycling agent
                           selection criteria (Reference 4)
- Density-voids analysis TAI MS-2   (Reference 8)
- Resilient modulus   ASTM D 4123   (Reference 11)
- Marshall parameters   ASTM D 1559   (Reference 11)

C. MACDILL AFB

1. Cores

- Bulk and apparent density ASTM D 2726   (Reference 11)
- Binder extraction   ASTM D 2172   (Reference 11)
- Binder recovery   ASTM D 1856 modified (Reference 5)
- Resilient modulus   ASTM D 4123   (Reference 11)
- Marshall parameters   ASTM D 1559   (Reference 11)

11

2. Core Binder

   • Same as Tyndall AFB

3. Core Aggregates

   • Gradation   ASTM C 136   (Reference 11)

4. RAP Binder

   • Viscosity   ASTM D 2170, D 2171, D 2281
                                                   (Reference 11)

   • Penetration   ASTM D 5   (Reference 11)
   • Composition   ASTM D 2007 modified (References 4,12)
   • Solubility   Heithaus modified   (References 4,6)

5. Rejected RAP Aggregates

   • Abrasion*   FM 1 T 096 (Appendix D and
                                                   (Reference 10)

6. Modifiers

   • Same as Tyndall AFB

7. Modifier/RAP Binder Blends

   • Same as Tyndall AFB

_____

* Test conducted by Dr. Edees,
  University of Florida, Gainsville.

12

D. HURLBURT FIELD

1. Cores

- Bulk and apparent density ASTM D 2726   (Reference 11)
- Binder extraction   ASTM D 2172   (Reference 11)
- Binder recovery   ASTM D 1856 modified (Reference 5)
- Resilient modulus   ASTM D 4123   (Reference 11)
- Marshall stability flow   ASTM D 1559   (Reference 11)

2. Core Binder

- Penetration   ASTM D 5   (Reference 11)
- Viscosity   ASTM D 2170, D 2171, D 3381
                                                  (Reference 11)
- Composition   ASTM D 2007 modified (References 4,12)
- Solubility   Heithaus modified   (References 4,6)

3. RAP Binder

- Viscosity   ASTM D 2170, D 2171, D 3381
                                                  (Reference 11)
- Penetration   ASTM D 5   (Reference 11)
- Composition   ASTM D 2007 modified (References 4,12)
- Solubility   Heithaus modified   (References 4,6)

4. Modifiers

- Viscosity   ASTM D 2170, and 2171 (Reference 11)
- Composition   ASTM D 2007 modified (References 4,12)
- Solubility   Heithaus modified   (References 4,6)

5. Modifier/RAP Binder Blends (Before RTFO)

- Viscosity   ASTM D 2170, D 2171, D 3381
                                                  (Reference 11)
- Penetration   ASTM D 5   (Reference 11)
- Rheology   Schweyer apparatus   (Reference 7)

- Ductility   ASTM D 113   (Reference 11)
- Composition   ASTM D 2007 modified (References 4,12)
- Solubility   Heithaus modified   (References 4,6)

6. Modifier/RAP Binder Blends (After RTFO)

- Penetration   ASTM D 5   (Reference 11)
- Ductility   ASTM D 113   (Reference 11)
- Viscosity   ASTM D 2171   (Reference 11)
- Composition   (References 4, 12)
- Solubility   (References 4, 6)

7. RAC

- Bulk and apparent density ASTM D 2726   (Reference 11)
- Binder extraction   ASTM D 2172   (Reference 11)
- Binder recovery   ASTM D 1356 modified (Reference 5)
- Resilient modulus   ASTM D 4123   (Reference 11)
- Marshall stability/flow   ASTM D 1559   (Reference 11)

E. DESCRIPTION

1. Tyndall AFB

a. Cores

The objectives of density testing of cores from Tyndall were to determine the level of compaction uniformity along the pavement. Thirty cores were divided into six groups of five representing approximately equal segments of the pavement. Density was evaluated by conventional one-way analysis of variance (ANOVA) to determine if a significant difference in density occurred along the pavement.

Binder and aggregate from five pairs of cores were evaluated as shown in the previous summary. These five pairs represented trafficked and nontrafficked pavement areas as shown below:

14

| | Core No. | |
Core Pair	Trafficked	Nontrafficked
1	12	24
2	14	16
3	22	46
4	26	28
5	50	56

Extractions of all core pairs was accomplished; however, not all pairs were evaluated. Viscosity and penetration were measured for Core Pairs 1, 2, and 3, and aggregate gradation was measured for Core Pairs 2, 4, and 5. Inference statistics were used to estimate differences between the parameters measured for the various pairs. Composition and solubility test procedures are modifications of published methods (Reference 6 and 12). The modifications are described in Appendices A and B.

b. Modifier/Core Binder Blends

Blends of core binder and each of the three modifiers were fabricated in the laboratory. All blends were fabricated according to the Chevron blend chart shown in Figure 4 to produce a resulting blend viscosity of 2000 ± 400 poises.

2. MacDill AFB

a. RAC Cores

Six-inch-diameter cores were obtained for NMERI by the base personnel from Phase A reconstruction. These cores were tested for bulk and apparent density prior to recoring to a more manageable 4-inch-diameter. The 4-inch specimens were evaluated for Marshall parameters and resilient modulus.

b. RAP

NMERI was supplied RAP from Phase B of the recycling project by base personnel. Since cores were obtained from different pavement features than from RAP, comparison of RAP properties with core properties is avoided.

15
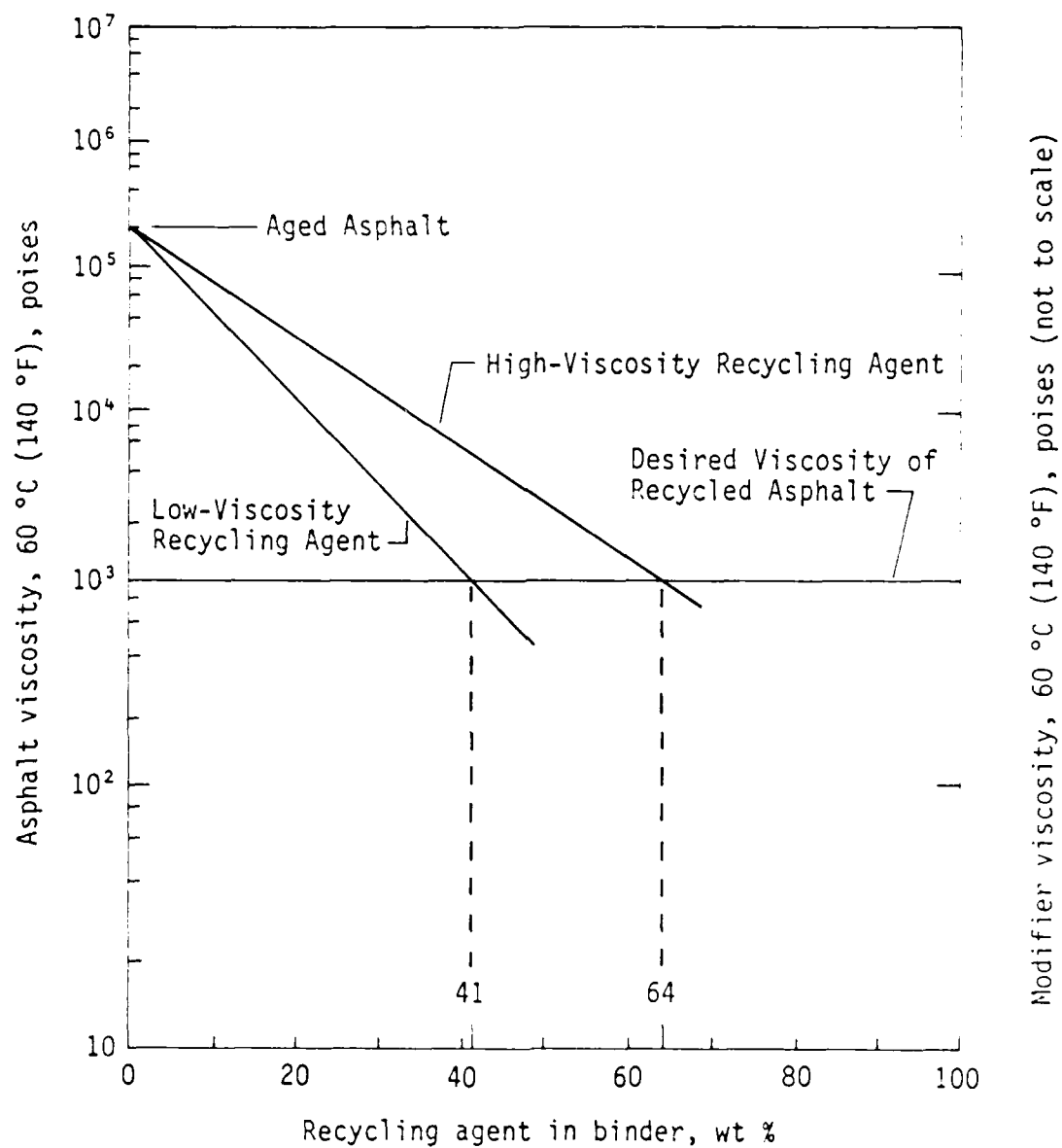
Figure 4. Percent Recycling Agent Required--Dependent On Viscosity
(Reference 9)

c. Modifier/RAP Binder Blends

Reclaimed RAP binders were blended with MBD-D through MBD-G so
that the resulting viscosity of the blend was 4000 ± 800 poises. Proportioning
of RAP binders and modifiers was done using the Chevron blend chart shown in
Figure 4.

3. Hurlburt Field

a. Modifier/RAP Binder Blends

Reclaimed RAP binders were blended with MBD-2D and MBD-2DD so
that the resulting viscosity of the blend was 2000 ± 400 poises. Proportioning
of RAP binders and modifiers was done using the Chevron blend chart shown in
Firgure 4.

b. RAC Cores

Two sets of cores are described in this report. The first set,
seven cores, was taken by the paving contractor and supplied to NMERI by base
personnel. The second set, 17 cores, was obtained and tested by the paving
contractor. The results of laboratory tests of these cores were supplied to
NMERI by base personnel.

17

SECTION IV
RESULTS

A. TYNDALL AFB

1. Cores

Six groups of five cores each were evaluated for bulk density. The results of this testing appear in Table 1. Cores are ordered in Table 1 from Group 1 to Group 6 beginning at the top of the column, i.e., Core Number 2 bulk density is 2.211; Core Number 4, 2.135, and so forth.

The ANOVA source table at the bottom of Table 1 indicates a highly insignificant F-statistic for groups. This indicates that no significant difference in bulk density is present between groups of cores.

Because of the small quantity of material available for testing, cores were combined so that enough binder would be available for testing. To do this, cores were paired from trafficked and nontrafficked pavement areas as previously stated. Extractions of the binder from these combined cores allowed further evaluation by viscosity, penetration, and gradation. Results of this work are shown in Tables 2 and 3 for certain pairs of cores. These results seem to indicate that physical properties for combined binders of trafficked and nontrafficked pavement regions are similar. Confidence intervals for the mean viscosity, penetration, percent binder, and gradation are shown in Tables 2 and 3. Note that all measured values lie within the 95 percent confidence limits for the mean of each parameter.

Because of the scarcity of binder, and because binder properties appeared reasonably uniform, the binder from all cores was mixed. The results of viscosity and penetration tests of this combined binder appear in Table 2.

2. Modifiers

Results of physical and chemical laboratory tests on the three Tyndall AFB modifiers are shown in Table 4. Some properties of the virgin AC-20 are included. Certain recommended limiting values (References 4 and Appendix A) are also shown for some of these test parameters to help identify

TABLE 1.   TYNDALL CORES, DENSITY ANOVA

			Group			
	1	2	3	4	5	6
	2.211	2.266	2.212	2.218	2.301	2.270
	2.135	2.228	2.209	2.280	2.218	2.260
	2.230	2.129	2.300	2.209	2.256	2.260
	2.230	2.327	2.241	2.210	2.234	2.340
	2.220	2.169	2.230	2.143	2.300	2.260
Sum =	11.026	11.119	11.192	11.060	11.309	11.390
Avg. =	2.205	2.224	2.238	2.212	2.262	2.278
n =	5	5	5	5	5	5
Σy^2 =	24.32	24.75	25.06	24.47	25.58	25.95

CT	=	150.06
SS Groups	=	0.02
SS Total	=	0.08
SS Error	=	0.06

ANOVA

Source	df	SS	MS	F	F.05
Group	5	0.0205	0.0041	1.750	2.62
Error	24	0.0563	0.0023		
Total	29	0.0769			

19

TABLE 2. TYNDALL CORE PAIR BINDER, PHYSICAL PROPERTIES

Test Parameter	Core Pair					Binder from core pairs 1-5
	1	2	3	4	5	
Viscosity, 140F, P	23600	24350	24550	n/a	n/a	24170
95% C.I. =		22900 to 25400				
Penetration, 77F, .01mm	17	13	13	n/a	n/a	13
95% C.I. =		9 to 20				
Binder, %	6.1	6.3	5.4	6.0	5.2	--
95% C.I. =			4.6 to 6.9			

Composition:

Saturates, %	No data	18.19
Asphaltenes, %	for	37.93
Aromatics, %	individual	15.2
Polars, %	cores	28.68
Polars ÷ Saturates		1.58
Asphaltenes+Saturates		56.12

Solubility:

Asphaltene Peptizability, (P_a)		0.61
Maltene Peptizing Power, (P_o)	No data	1.13
State of Peptization, (P)	for	2.92
Limiting Dilution Ratio, (X_{min})	individual	1.92
Limiting Titrant Volume, (T_o)	cores	2.18
Waxman's Cotangent		1.45

TABLE 3. TYNDALL CORE PAIR AGGREGATE, PHYSICAL PROPERTIES

Sieve Size	% Passing Core Pair			% Passing 95% C.I.	Spec * % Passing
	2	4	5		
3/4"	100	100	100	100	100
1/2"	97	96	97	97±1.4	84-100
3/8"	94	91	91	92±4.3	75-93
4	65	66	67	66±2.5	59-73
8	30	45	51	45±26.9	46-60
16	27	32	36	31±11.2	34-48
30	18	22	24 -	22±7.6	24-38
50	12	14	15	13±3.8	15-27
100	7	7	7	7±0	8-18
200	2.5	2.7	1.9	2.4±1.0	3-6

S.G. +3/8"=   2.732   2.746   2.732
S.G. -3/8"=   2.773   2.808

* From construction specifications, McDill AFB recycling project, MCD 65-8,
   by A. Ravicher, P.E.

21

TABLE 4. TYNDALL MODIFIER PROPERTIES

Property	Modifier				Limits, Ref in parenths
	MBD-A	MBD-B	MBD-C	AC-20	
before RTFO:					
Viscosity, 100F, P	22594	8393	1545	-	-
Viscosity, 140F, P	581	276	65	1815	1.0 (min)(3)
Viscosity, 212F, cSt	1470	957	342	-	15.0 (min)(3)
Viscosity, 275F, cSt	-	-	-	336	-
Slope LogVis/LogTemp	8.86	8.10	7.10	-	-
after RTFO:					
Viscosity, 140F, P	1166	480	87	5254	-
Penetration, 77F, 0.01mm	-	-	-	42	-
Weight Loss, %	0.29	0.26	0.17	2.89	1.0 (max)(3)
Viscosity Ratio,	2.06	1.74	1.34	-	3.0 (max)(3)(13)
Composition:					
Saturates, %	23.22	22.9	22.9	-	-
Asphaltenes, %	17.21	13.63	9.77	-	-
Aromatics, %	16.96	19.44	25.68	-	-
Polars, %	42.6	44.04	41.65	-	-
Polars ÷ Saturates	1.83	1.92	1.82	-	0.50 (min)(3)
Asphaltenes+Saturates	40.43	36.53	32.67	-	40 (max)(App. A)
Solubility:					
Asphaltene Peptizability, (P_a)	0.66	0.68	0.8	-	-
Maltene Peptizing Power, (P_o)	1.25	1.21	1.06	-	$3.0 \pm 0.5 (min)$
State of Peptization, (P)	3.61	3.8	5.22	-	(App. A)
Limiting Dilution Ratio, (X_{min})	2.61	2.8	4.22	-	-
Limiting Titrant Volume, (T_o)	2.91	2.08	4.96	-	-
Waxman's Cotangent	1.75	2.48	3.55	-	-

Note: Viscosity at 100F and 140F by ASTM D2171
Viscosity at 212F and 275F by ASTM D2170

which of the modifiers tested appears the most desirable for recycling purposes.

Note the viscosity at 140 °F for the recycling agents. Each satisfies requirements associated with AC-5, AC-2.5, and AC-0.5 for MBD-A, MBD-B, and MBD-C, respectively.

Some characteristics of modifiers that are important to the recycled pavement are shown below.

	MBD-A	MBD-B	MBD-C	AC-20
• Slope of viscosity/temperature curve	202	75	14	13
• Weight loss, percent	0.29	0.26	0.17	--
• Viscosity ratio	2.06	1.74	1.34	2.89
• Asphaltenes + Saturates	40.4	36.5	32.7	--
• State of peptization	3.61	3.80	5.22	--

The slope of the viscosity/temperature curve is a measure of the temperature susceptibility of a material. Low-temperature susceptibility is desirable for asphalt binders; therefore it should be an important property for recycled binders. Since it is logical that the temperature susceptibility, and all other properties, of the recycled binder (reclaimed pavement binder + modifier) would be influenced by the modifier, a modifier with low-temperature susceptibility appears desirable. A minimum value of temperature susceptibility of a modifier cannot be established within the scope of this effort.

Weight loss is an indication of volatilization of a material. Volatiles are often associated with the maltene phase of an asphalt. Maltenes are necessary to help prevent early hardening of an asphalt. Therefore, low weight loss is desirable.

Viscosity ratio is a measure of the increase in hardening of an asphalt after it is heated in the presence of air. A low viscosity ratio is desirable because it indicates that the asphalt will become brittle at a slower rate than an asphalt with a high viscosity ratio.

23

The quantity of asphaltenes and saturates affects the above properties in a directly proportional manner except for the state of peptization. Therefore the lower the value of this parameter, the less likely the asphalt will harden as it ages.

State of peptization is a measure of the dispersion of the asphaltenes in asphalt. High dispersion is desirable because hardening is accelerated as asphaltenes coalesce.

Modifier MBD-C appears to be the most desirable candidate of the three evaluated for use as a recycling agent, based on the above.

3. Modifier/Core Binder Blends

The three recycling agents were each blended with extracted and recovered asphalt from the cores. Each blend contained varying quantities of recycling agent so that viscosity of the blend lies within viscosity tolerances specified at 140 °F in ASTM D 3381 for AC-20. Physical and chemical properties of these blends are shown in Tables 5 and 6. Blends are identified with the numeral 6 added to the modifier identification, as MBD-B6, for the modifier, MBD-B, blended with core binder.

A comparison of blend properties may indicate which blended material will have the most desirable performance in the field. Comparing properties as before:

	MBD-A6	MBD-B6	MBD-C6
• Slope of viscosity/temperature curve	15.3	14.9	12.4
• Weight loss, percent	-	-	-
• Viscosity ratio	2.07	2.04	1.70
• Asphaltenes + Saturates*	43.42	41.10	42.73
• State of peptization *	2.56	2.37	3.45
• Percent modifier	63	50	33

----

* After RTFO aging

TABLE 5. TYNDALL MODIFIER/CORE BINDER, PHYSICAL PROPERTIES

| Property | Modifier/Core Binder Blend | | | AC-20 Spec |
	MBD-A6	MBD-B6	MBD-C6	ASTM D3381
before RTFO:				
Viscosity, 77F, P x E3	252	267	204	-
Viscosity, 140F, P	2073	2019	1676	1600-2400
Viscosity, 275F, cSt	336	326	304	210 (min)
Slope LogVis/LogTemp	9.51	9.52	9.35	-
Penetration, 77F, 0.01mm	60	60	69	40 (min)
Penetration, 39.2F, 0.01mm	33	50	63	-
Ductility, 60F, cm	100+	100+	100+	-
after RTFO:				
Viscosity, 140F, P	4286	4127	2844	-
Viscosity Ratio,	2.07	2.04	1.7	-
Penetration, 77F	41	43	54	-
Penetration Retained, %	68	72	78	-
Ductility, 60F, cm	13	25	61	-
Modifier, % by blend weight required for 2000P viscosity	63	50	33	

Note: Viscosity at 140F by ASTM D2171
       Viscosity at 275F by ASTM D2170
       Viscosity at 77F by Schweyer Rheometer (9)

TABLE 6. TYNDALL MODIFIER/CORE BINDER BLEND, CHEMICAL PROPERTIES

| | Modifier/Core Binder Blend | | | | | |
| | MBD-A6 | | MBD-B6 | | MBD-C6 | |
Property	Before RTFO	After RTFO	Before RTFO	After RTFO	Before RTFO	After RTFO
Composition:						
Saturates, %	19.49	20.80	19.98	20.49	19.89	19.30
Asphaltenes, %	21.10	22.62	21.18	20.61	22.54	23.43
Aromatics, %	17.08	15.65	17.35	16.93	17.49	18.11
Polars, %	42.33	40.93	41.52	41.97	40.08	39.16
Polars ÷ Saturates	2.17	1.97	2.08	2.05	2.02	2.03
Asphaltenes+Saturates	40.59	43.42	41.16	41.10	42.43	42.73
Solubility:						
Asphaltene Peptizability, (P_a)	0.60	0.66	0.62	0.59	0.52	0.51
Maltene Peptizing Power, (P_o)	1.24	0.87	1.06	0.96	1.48	1.70
State of Peptization, (P)	3.12	2.56	2.77	2.37	3.07	3.45
Limiting Dilution Ratio, (X_{min})	2.12	1.56	1.77	1.37	2.07	2.45
Limiting Titrant Volume, (T_o)	2.06	2.00	1.93	1.51	2.28	2.26
Yaxman's Cotangent	1.57	1.74	1.53	1.39	0.97	0.95

After modification, the core binder displays remarkably similar properties independent of the modifier used. However, the above data suggest modifier MBD-C produces the most desirable properties when blended with this particular core binder due to lowest percentage, highest state of peptization, and lowest viscosity ratio.

4.   Recycled Asphalt-Concrete (RAC)

Virgin aggregates, modifier MBD-C, and virgin AC-20 asphalt were added to aggregates and asphalt from pavement cores to produce RAC. Virgin AC-20 was added in quantities so that the desired 5.0, 5.5, and 6.0 percent binder-by-mix weight would result. Core mixtures and virgin aggregate were mixed at ratios of 30:70, 40:60, and 50:50 by weight to produce RAC mixtures having the target gradation shown in Table 7. This target is obtained by using the average gradation of the three core pairs (shown in Table 3) from the 3/4-inch seive to the Number 4 sieve. The midband of the specification shown in Table 3 is used for the Number 8 to Number 200 sieves.

Three different virgin aggregate gradations were required to modify the average core gradation so that the target gradation would result. The three gradations used are shown in Table 7.

Modifier MBD-C was added to core mixtures so that the resulting blended binder, after addition of AC-20 to bring the total binder to the desired 5.0, 5.5, or 6.0 percent values, contained 33 percent MBD-C by modifier and core binder weight.

Marshall mixture design procedures were used to determine optimum binder content of the RAC mixtures. Three specimens each at three binder contents were prepared for each RAC mixture. To produce the three different binder contents, some additional virgin AC-20 was added to each of the mixes. Therefore, the RAC mixtures contained three types of binders: (1) the core binder, (2) MBD-C, and (3) virgin AC-20.

27

TABLE 7. AGGREGATES USED IN TYNDALL RAC

Sieve Size	Core Pairs, Avg. % Passing	Virgin Aggregate % Passing 30:70 Mixes	Virgin Aggregate % Passing 40:60 Mixes	Virgin Aggregate % Passing 50:50 Mixes	Target Gradation, % Passing
3/4"	100	100	100	100	100
1/2"	97	97	97	97	97
3/8"	92	92	92	92	92
4	66	66	66	66	66
8	45	60	54	57	53
16	31	45	39	42	41
30	22	30	27	29	28
50	13	25	19	22	21
100	7	15	12	14	13
200	2.4	5	3	5	4.5

Results of laboratory testing of the three types of RAC mixtures are presented in Table 8. A graphical representation of this data is presented in Figures 5 through 10.

The RAC mixtures satisfy all Air Force AFM 88-6 mix design criteria except air voids. Required are air voids between 3 and 5 percent. The 50:50 RAC mixture at 6 percent binder content is the only mixture evaluated which meets these criteria. Properties of the mixture at 6 percent binder are as follows:

- Marshall stability, lb   2300
- Marshall flow, 0.01 in   12
- Air voids, percent   4.0
- VMA, percent   17.9
- Unit weight, lb/ft^3 150.5
- Resilient modulus, lb/in^2 $(x\ 10^3)$ 118

The high voids in RAC mixes, 30:70 and 40:60, are evidently produced by the higher virgin aggregate percentage. Assuming gradation is not changed, these high voids could be reduced by increasing binder content above 6 percent.

5. Oven-Aged RAC

Nine Marshall test specimens were prepared using the 50:50 RAC material described above at 6 percent binder. These specimens were oven aged at 140 °F for 0, 7, 28, 74, and 123 days and evaluated for Marshall properties and resilient modulus. Testing was conducted so that nine specimens were tested at 0 days for resilient modulus (nondestructive), and one of the nine specimens was tested for Marshall parameters (destructive). Thereafter, the eight remaining specimens were tested at 7 days for resilient modulus, and one of the eight specimens for Marshall properties. In this way, all specimens were finally tested after 123 days. The results are shown in Table 9 and plotted in Figures 11 and 12.

29

TABLE 8. TYNDALL RAC PROPERTIES

RAP/Virgn Aggregate Ratio	Binder, % by mix weight	Marshall Stability, lbs	Marshall Flow, .01 in	Air Voids, %	VMA, %	Unit Weight, pcf	Resilient Modulus, psi × 1000
30/70	5.0	2130	10	8.7	20.5	144.50	85.30
	5.5	1900	11	6.8	19.9	146.40	67.60
	6.0	1700	11	6.1	19.9	147.30	66.50
40/60	5.0	2560	10	8.6	19.3	146.70	135.90
	5.5	2570	11	6.4	19.0	148.10	130.60
	6.0	2200	12	5.4	19.3	148.30	103.60
50/50	5.0	2400	11	7.0	18.4	148.30	254.70
	5.5	2700	11	5.1	18.1	149.80	227.90
	6.0	2300	12	4.0	17.9	150.50	117.80
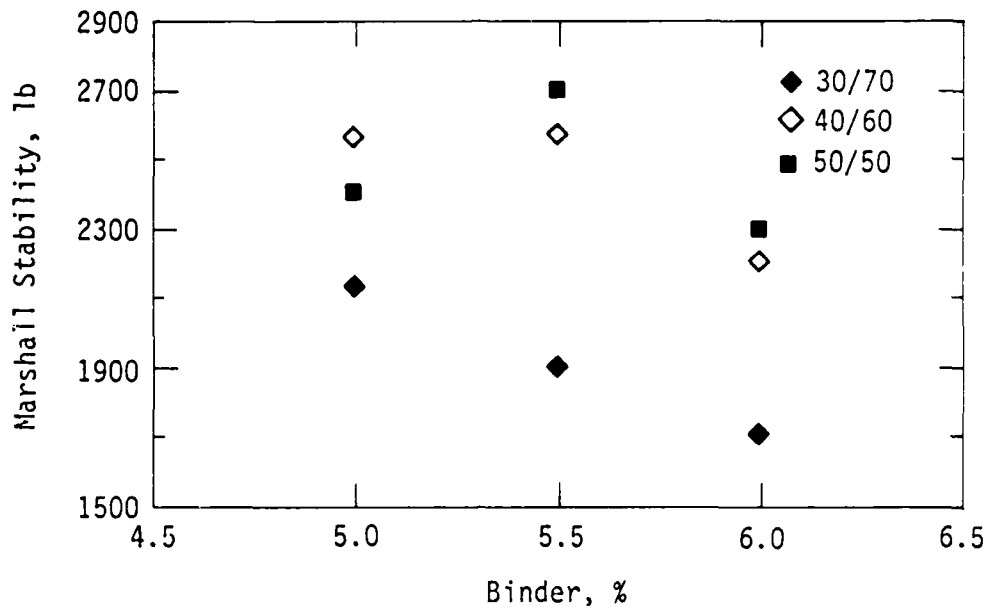
Figure 5. Tyndall AFB RAC, Marshall Stability versus Percent Binder.
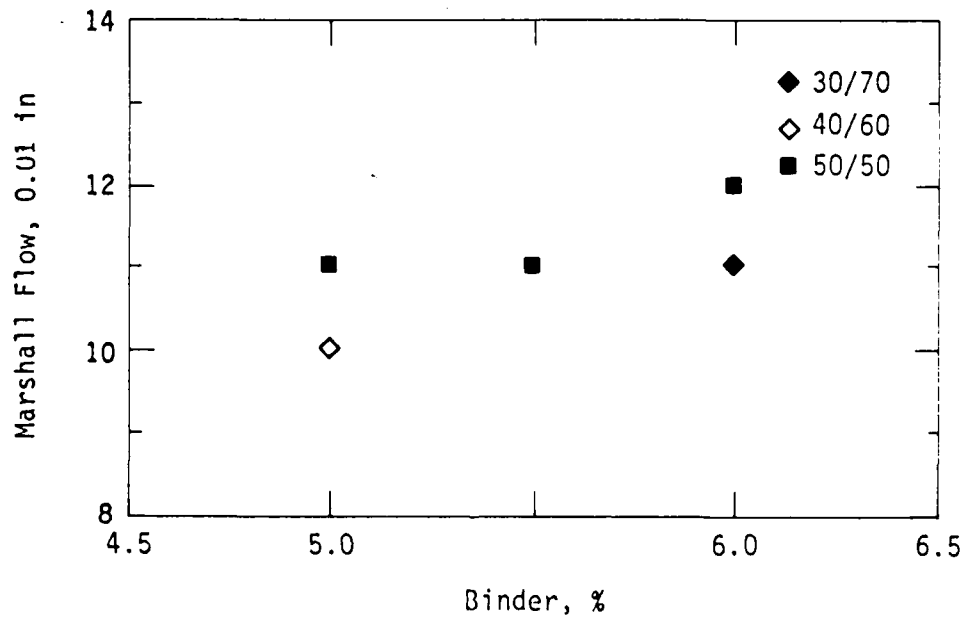
Figure 6. Tyndall AFB RAC, Marshall Flow versus Percent Binder.
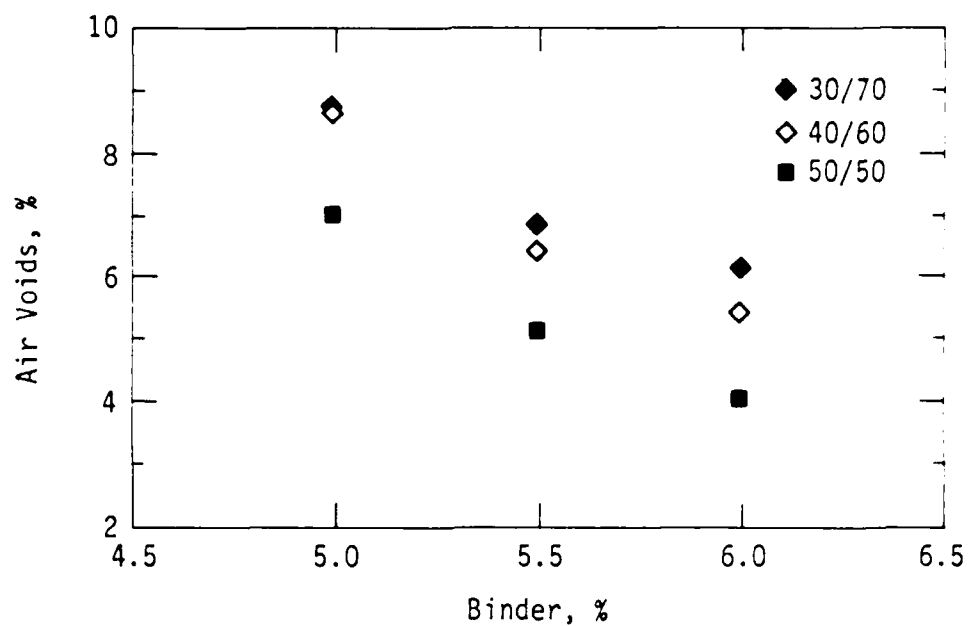
Figure 7. Tyndall AFB RAC, Air Voids versus Percent Binder.
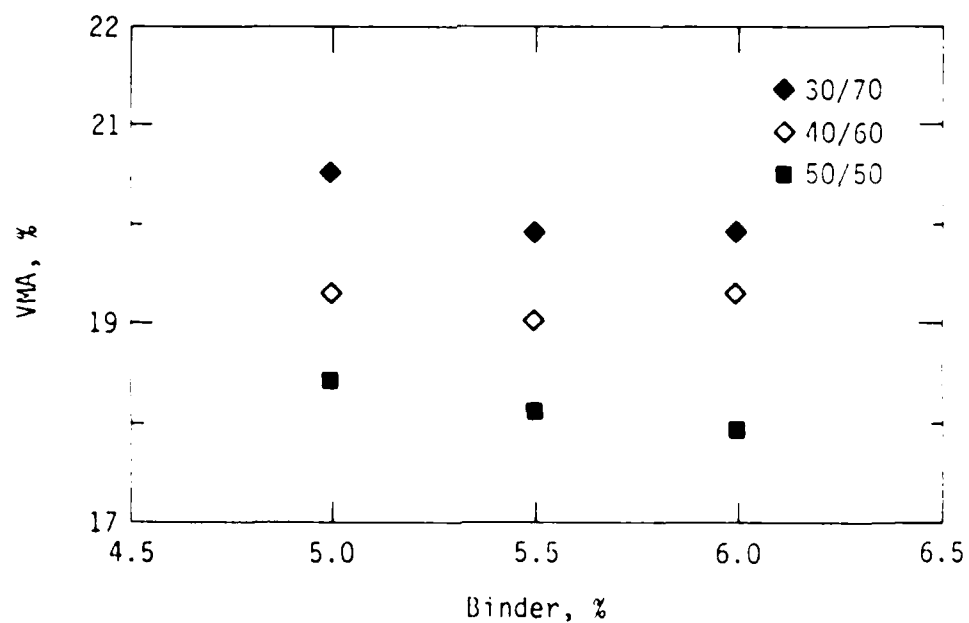
Figure 8. Tyndall AFB RAC, VMA versus Percent Binder.
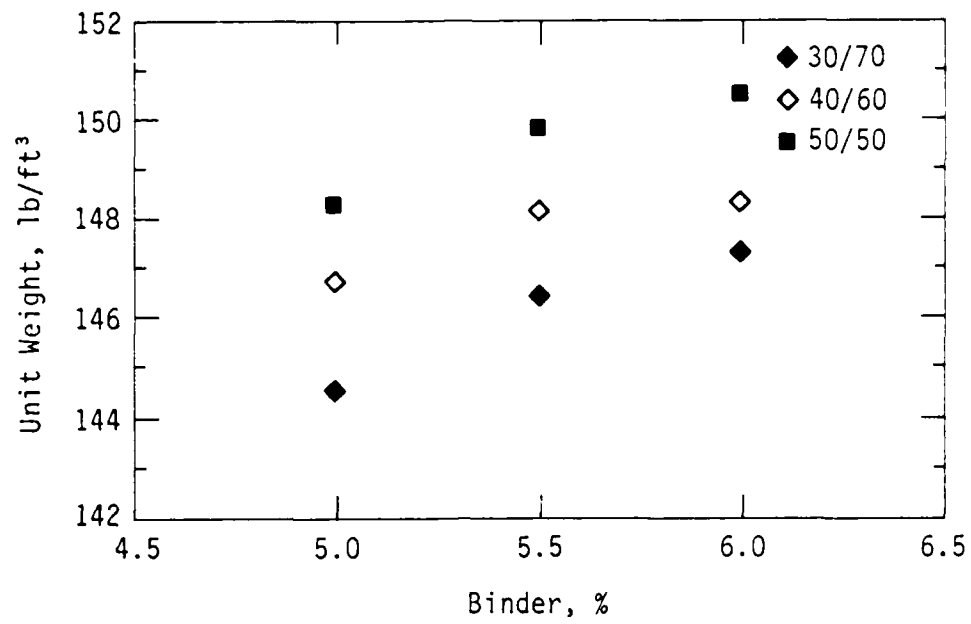
Figure 9. Tyndall AFB RAC, Unit Weight versus Percent Binder.
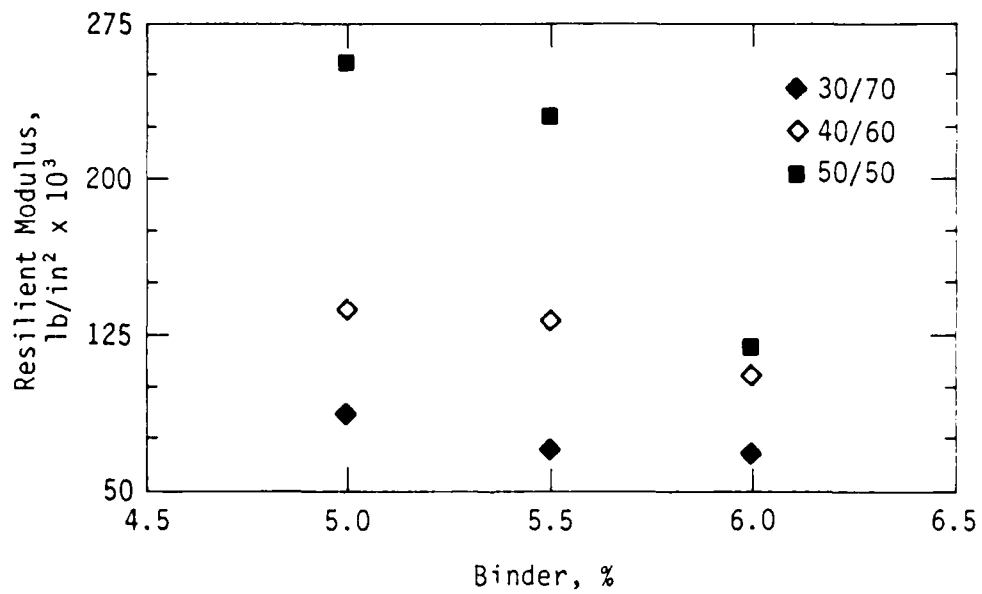
Figure 10. Tyndall AFB RAC, Resilient Modulus versus Percent Binder.

TABLE 9. TYNDALL RAC PROPERTIES AFTER OVEN AGING

Aging Period, days	Unit Weight, pcf	Air Voids, %	Marshall Stability, lbs	Marshall Flow, .01 in	Resilient Modulus, psi × 1000
0	145.1	7.1	2889	14	282
7	144.8	6.0	2208	12	159
28	145.0	5.6	3164	13	194
74	145.3	4.4	3221	12	205
123	149.1	4.2	3252	13	333
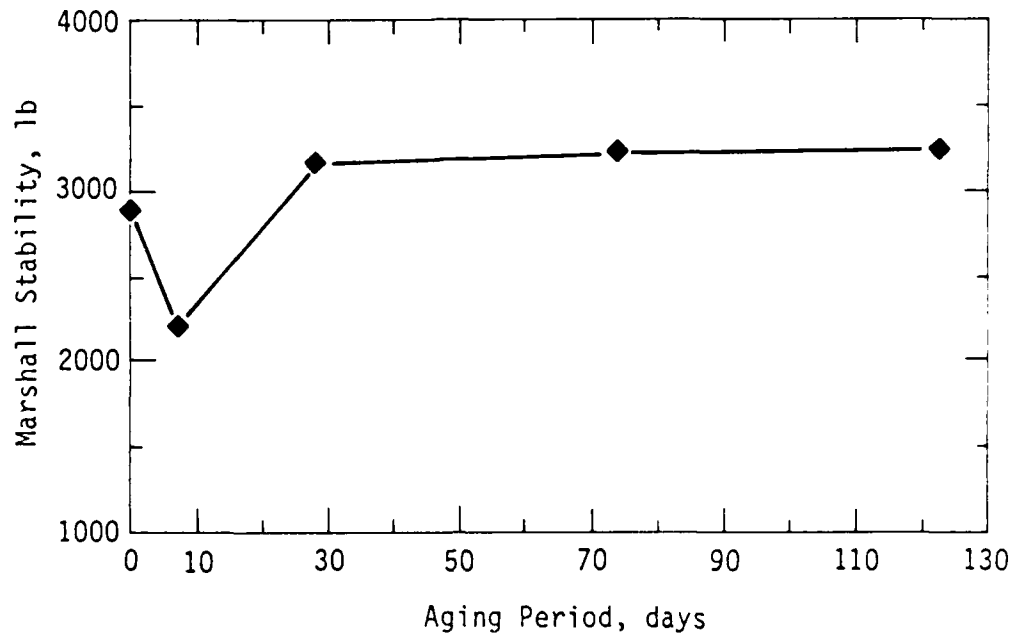
Figure 11. Tyndall AFB 50:50 RAC Marshall Properties.
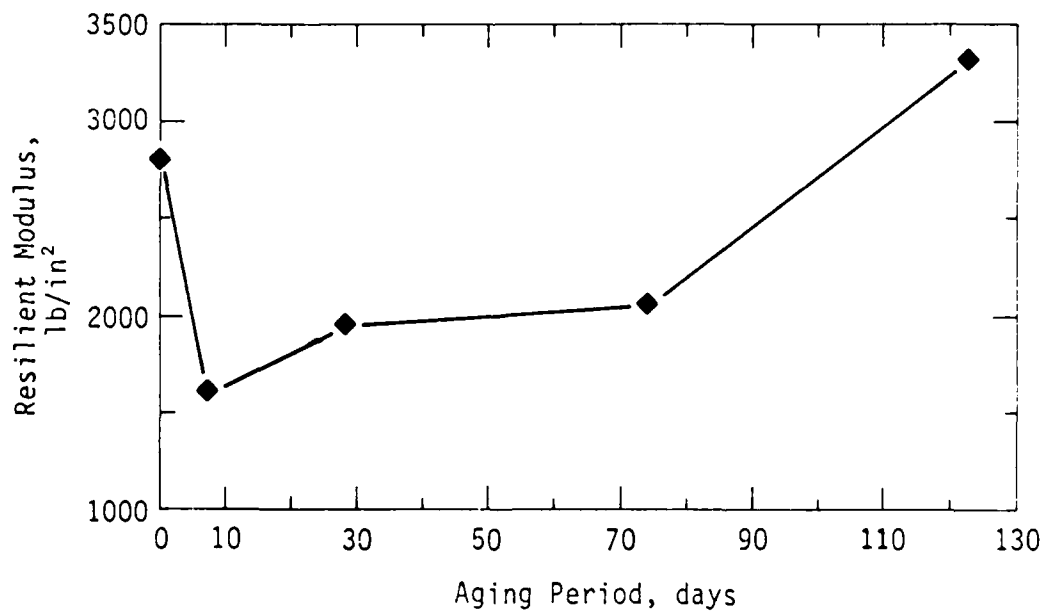
Figure 12. Tyndall AFB 50:50 RAC Resilient Modulus.

An interesting trend occurs for Marshall stability and resilient modulus as aging progresses. There is a loss of stability and a lowering of modulus at 7 days aging, followed by an increase in both properties as aging continues. This is evidently due to the softening ability of the modifier. Initially, at 0 days, no softening has occurred, and properties are unaffected by the modifier. At 7 days, softening of the reclaimed binder occurs, and stability and modulus decline. As aging progresses, hardening of the blended binder occurs, and stability and modulus begin to increase. Marshall stability seems to become constant at about 28 days. Resilient modulus has not leveled off at 123 days.

Binders were extracted after each aging period and evaluated for physical and chemical properties. Results are shown in Table 10. The results indicate viscosity to be the only parameter which measures significant changes in binder properties as aging progresses. A fourfold increase occurs in 140 °F, viscosity between 0 and 74 days. By contrast, the next most significant change in any parameter is to T_0 with a 1.44 times decrease. This result suggests that identification of aging by chemical means may appear to be inconclusive.

However, a contrasting situation can be cited from the technical literature. In a study documented in Reference 5, two California asphalts, coastal and valley, were investigated as follows. The two asphalts had an original consistency of AC-40 and each was air-blown to about 50,000 poises viscosity at 140 °F. The coastal asphalt reached a blown mean viscosity of 45,559 poises and valley asphalt a value of 49,508 poises at 140 °F. The resulting aging indices were 10.6 for coastal and 12.1 for valley. Based on these physical test results one would choose coastal asphalt because it hardened the least amount. NMERI analyzed these materials for solubility and found the following for the state of peptization (P): coastal soft (AC-40) and coastal air-blown had 3.18 and 2.98; valley soft (AC-40) and valley air-blown had 5.19 and 4.58. Using the P-values, the valley asphalt is more homogeneously dispersed than the coastal asphalt and hence valley asphalt would be a better choice than the coastal asphalt. This is because valley asphalt maintained a higher state of internal dispersion than coastal asphalt due to air blowing. The significance of this argument is that, although changes in physical properties may be many times the changes in chemical properties of an asphalt system during aging, the choice to be made may be influenced by results of a chemical analysis.

36

TABLE 10.   TYNDALL RAC BINDER PROPERTIES

	Oven Aging Period, days				
	0	7	28	74	123
Viscosity, 140F, P	2072	3597	-	7909	6605
Penetration, 77F, 0.01mm	-	-	-	33	38
Penetration, 39.2F, 0.01mm	-	-	-	16	18
Composition:					
Saturates, %	21.61	22.26	-	21.63	20.81
Asphaltenes, %	22.68	22.96	-	24.84	24.84
Aromatics, %	14.61	14.2	-	13.18	13.51
Polars, %	41.1	40.58	-	40.36	40.84
Polars ÷ Saturates	1.9	1.82	-	1.87	1.96
Asphaltenes+Saturates	44.29	45.22	-	46.47	45.65
Solubility:					
Asphaltene Peptizability, (P_a)	0.64	0.62	-	0.64	0.65
Maltene Peptizing Power, (P_o)	1.17	1.14	-	1.05	1.01
State of Peptization, (P)	3.27	3.04	-	2.87	2.85
Limiting Dilution Ratio, (X_{min})	2.27	2.03	-	1.87	1.85
Limiting Titrant Volume, (T_o)	2.44	1.98	-	1.69	1.85
Waxman's Cotangent	1.72	1.68	-	1.68	1.78

B.   MACDILL AFB

1.   RAC Cores

Thirteen of the fourteen 6-inch cores were evaluated for thickness
and density prior to recoring to 4-inch diameters. The 4-inch versions of the
6-inch cores were then evaluated for Marshall properties and resilient
modulus. Results of this work appear in Table 11. The stability data in this
table are presented for general information purposes only.

Fewer cores were supplied from MacDill than from Tyndall;
consequently the type of analysis done for Tyndall core density was not
possible for MacDill. However, 95 percent confidence limits have been
developed for thickness and density for the 13 cores, and are reported at the
bottom of Table 11. Inspection of these limits and the core data suggest
significant variability may be present along Taxiway 7 with respect to
thickness and bulk density. Six of the thirteen cores are outside the
thickness limits, and seven of the thirteen cores are outside the density
limits. Because the locations where various modifiers were used are
unavailable, the variability in core properties cannot be compared to modifier
properties.

2.   RAC Core Aggregates

Seven of the fourteen cores were chosen at random, extracted for the
asphalt binder, and sieve analysis run on the resulting aggregates. These
results are shown in Table 12. The average sieve analysis for the seven cores
is also shown with 95 percent confidence limits applied. Note that aggregate
gradations for cores 4b and 4c are consistently outside confidence limits
applied to the average gradation.

3.   RAC Core Binder

The seven cores not evaluated for aggregate gradation were also
extracted and binders evaluated for certain physical and chemical properties.
The results of physical testing appear in Table 13. Chemical test results are
summarized in Table 14.

38

TABLE 11. MACDILL CORE PROPERTIES

	6 inch Cores:			4 inch Cores:			
Core No.	Thickness, in	Bulk Density, pcf	Marshall Stability, lbs	Marshall Flow, .01 in	Resilient Modulus Dry, psi × 1000	Wet, psi × 1000	Retained Modulus, %
1a	2.14	133.3	860	27	584	729	125
1b	2.19	136.4	1150	20	412	688	167
2a	2.25	140.4	damaged	damaged	234	231	99
2b	2.21	138.2	860	18	161	245	153
2c	2.10	130.9	710	17	195	326	167
3a	2.53	157.7	1940	25	582	807	139
3b	2.48	154.6	2350	24	477	728	153
4a	2.28	142.1	1210	24	387	646	167
4b	2.13	132.8	damaged	damaged	94	151	161
5a	1.93	120.6	680	15	229	259	113
5b	1.74	108.6	520	16	218	474	218
6a	1.93	120.6	1350	19	400	550	138
6b	1.88	117.5	1780	18	714	685	96

95%
Confid 2.00 to 124.9 to
Interval 2.28 141.90

TABLE 12. MACDILL SELECTED CORE GRADATIONS

Sieve Size	1a	2a	3a	4b	4c	6a	6b	Avg. % Passing ±95% C.I.	Spec % Passing
3/4"	100	100	100	100	100	100	100	100	100
1/2"	100	99	99	99	99	98	98	99±0.64	84-100
3/8"	93	90	93	98	96	91	93	93±2.6	75-93
4	67	70	67	86	80	67	70	72±7.0	59-73
8	51	54	50	69	64	50	52	56±7.1	46-60
16	44	47	43	56	53	42	43	47±5.1	34-48
30	34	36	34	46	43	34	33	37±4.8	24-38
50	23	24	23	31	29	24	23	25±3.1	15-27
100	11	11	12	9	9	12	12	11±1.2	8-18
200	5.9	6.3	6.4	3.9	4.7	6.8	7.2	5.9±1.1	3-6

The column header "% Passing Core No." spans columns 1a through 6b.

TABLE 13. MACDILL CORE BINDER PHYSICAL PROPERTIES

Test Parameter	Core No. 1a	1b	2a	2b	2c	3a	3b	4a	4b	4c	5a	5b	6a	6b	Control[a]
Viscosity, 140F, Px1000	19.4	13.8	4.2	8.5	8.7	7.7	26.5	29.7	5.7	5.3	16.3	39.3	19.7	4.9	66.7
95% C.I. =	9.0 to 27.7														
Penetration, 77F, .01mm	27	31	51	40	35	42	27	23	43	43	-	22	29	54	9
95% C.I. =	27 to 41														
Penetration, 39.2F, .01mm	14	16	23	19	17	22	16	13	19	19	-	11	16	28	0
95% C.I. =	15 to 21														
Binder, %	5.8	5.9	5.9	5.9	6.3	6.1	5.8	5.9	8.0	6.5	5.9	10.8	5.8	6.1	6.5
95% C.I. =	5.8 to 7.2														

[a]The Control RAP was taken from Phase B of the construction. The cores were from another area, Phase A.

41

TABLE 14. MACDILL CORE BINDER CHEMICAL PROPERTIES

Test Parameter	Core No.														
	1a	1b	2a	2b	2c	3a	3b	4a	4b	4c	5a	5b	6a	6b	Control
Composition:															
Saturates, %	14.6	14.7	15.0	15.2	14.4	14.9	14.7	14.7	13.6	13.5	14.6	14.2	15.0	14.0	12.6
Asphaltenes, %	34.7	32.1	30.7	30.9	31.4	32.5	36.0	34.4	29.4	30.4	35.1	35.3	32.4	30.1	37.5
Aromatics, %	14.5	14.6	13.5	14.4	13.7	11.9	13.7	13.6	16.2	14.5	13.4	13.1	14.3	14.0	12.6
Polars, %	36.2	38.6	40.9	39.5	33.7	40.8	35.6	37.3	40.8	41.6	36.9	37.4	38.4	41.4	33.5
Polars + Saturates	2.48	2.63	2.73	2.60	2.34	2.74	2.42	2.54	3.00	3.08	2.53	2.63	2.56	2.96	2.66
Asphaltenes+ Saturates	49.3	46.8	45.7	46.1	45.8	47.4	50.7	49.1	43.0	43.9	49.7	49.5	47.4	44.1	50.1
Solubility:															
Asphaltene Peptizability, (P_a)	0.61	0.61	0.60	0.62	0.63	0.60	0.55	0.58	0.63	0.63	0.56	0.60	0.61	0.61	0.59
Maltene Peptizing Power, (P_o)	1.14	1.12	1.29	1.23	1.14	1.17	1.81	1.56	1.25	1.29	1.41	1.1	1.21	1.2	1.17
State of Peptization, (P)	2.95	2.88	3.19	3.24	3.07	2.89	4.00	3.69	2.41	2.46	2.19	1.75	2.13	2.06	1.85
Limiting Dilution Ratio, (X_{min})	1.95	1.88	2.19	2.24	2.07	1.89	3.00	2.69	2.41	2.46	2.19	1.75	2.13	2.06	1.85
Limiting Titrant Volume, (T_o)	2.10	1.90	2.23	2.17	2.05	1.92	2.94	2.76	2.33	2.37	2.13	1.78	2.14	2.12	2.01
Waxman's Cotangent	1.52	1.58	1.46	1.66	1.70	1.47	1.24	1.33	1.76	1.73	1.29	1.45	1.59	1.53	1.35

42

Physical properties appear to be measuring variability between core binders as shown by viscosity and penetration test results. Ten of 14 core binders have viscosity values outside the 95 percent confidence limits, and six of the 14 have penetration values, both 77 °F and 39 °F, lying outside these limits. This is not a surprising result since four types of modifiers were used as recycling agents on this pavement. Unfortunately, no data were supplied NMERI regarding specific locations of the four agents used. Therefore no conclusions can be offered relative to the cause of high variability in physical properties other than this result is probably caused by changes in agents.

The properties of the RAP binder are shown in both Tables 13 and 14, denoted as "Control."

Binder content is reasonably consistent. Two measured values lie outside the 95 percent confidence limits.

Chemical properties shown in Table 14 can be compared to statistics for each of these parameters shown in Table 15. Confidence limits have been prepared for each parameter so that judgment of variability between cores can be made. Note that the differences evident in physical properties are absent for chemical properties. This evidence suggests that the chemical parameters shown here are not sensitive to differences in the core binders, although physical test results indicate a significant difference exists.

4.   RAP Binder

Binder extracted from RAP of Phase B was evaluated for physical and chemical properties. The results appear in Tables 13 and 14 under the column heading "Control." These results appear in these tables for convenience only. Since RAP binder comes from Phase B and RAC core binders are from Phase A, a direct comparison is not possible. To do so would require assuming RAP binder from Phase A has the same properties as RAP binder from Phase B.

5.   Rejected RAP Aggregates

Gray slag aggregates from Phase B which were not used in the recycling effort were evaluated for abrasion resistance using the modified Los Angeles abrasion test (Appendix D). Values obtained for these materials are shown below.

TABLE 15.   MACDILL CORE BINDER CHEMICAL PROPERTY STATISTICS

Test Parameter	Average All Cores	Partial Σ sq dif	Partial Σ sq dif	Std Dev, s	$t \times s/(\sqrt{n})$	95% Confidence Lower Limit	Upper Limit
Composition:							
Saturates, %	14.5	6.32	0.76	2.53	1.46	13.05	15.96
Asphaltenes, %	32.5	75.97	6.19	8.74	5.05	27.48	37.58
Aromatics, %	14.0	12.82	0.82	3.59	2.07	11.88	16.03
Polars, %	38.5	90.36	6.72	9.53	5.50	33.00	44.01
Polars + Saturates	2.66	0.59	0.01	0.77	0.44	2.22	3.10
Asphaltenes+ Saturates	47.0	76.66	2.72	8.77	5.06	41.98	52.10
Solubility:							
Asphaltene Peptizability, (P_a)	0.60	0.01	0.00	0.09	0.05	0.55	0.65
Maltene Peptizing Power, (P_o)	1.28	0.46	0.03	0.68	0.39	0.89	1.67
State of Peptization, (P)	2.78	5.86	0.39	2.43	1.40	1.38	4.18
Limiting Dilution Ratio, (X_{min})	2.21	1.45	0.11	1.21	0.70	1.51	2.91
Limiting Titrant Volume, (T_o)	2.21	1.23	0.10	1.11	0.64	1.57	2.85
Waxman's Cotangent	1.52	0.34	0.03	0.59	0.34	1.18	1.86

Sieve Fraction Tested	Loss on Number 50 Sieve* After Abrasion, %	Maximum Permitted, %
No. 4 to No. 16	25	18
No. 16 to No. 30	39	18

6. Modifiers

Results of physical and chemical laboratory tests on the four modifiers
evaluated from MacDill AFB are shown in Table 16. Certain recommended limit-
ing values (References 4 and Appendix A) are also shown for some of these test
parameters to help identify which of the modifiers tested appears the most
desirable for recycling purposes.

Characteristics of modifiers discussed earlier for Tyndall materials
which should also be important to the recycled pavement at MacDill are shown
below.

	MBD-D	MBD-E	MBD-F	MBD-G
• Slope of viscosity/temperature curve		Not Determined		
• Weight loss, percent	-	-	0.77	0.47
• Viscosity ratio	2.27	2.21	2.09	1.91
• Asphaltenes + Saturates	38.65	38.28	38.63	38.03
• State of peptization	3.75	3.77	3.22	3.45

These modifiers cannot be judged precisely like the Tyndall modifiers because
temperature susceptibility and weight loss were not determined for all
materials. Temperature susceptibility could not be determined because no two
equivalent temperatures were used to evaluate all modifiers. Weight loss was
not measured for the AC-10 and AC-20 because of an insufficient supply of
material.

---

*No correlation has been made between modified Los Angeles abrasion results
 and ASTM C 535.

TABLE 16. MACDILL MODIFIER PHYSICAL AND CHEMICAL PROPERTIES

Property	Modifier				Limits, Ref
	MBD-D	MBD-E	MBD-F	MBD-G	in parenths
before RTFO:					
Viscosity, 39.2F, P x E+06	11.4	72.1	-	-	-
Viscosity, 77F, P x E+06	0.51	1.98	-	-	-
Viscosity, 100F, P	-	-	3491	4718	-
Viscosity, 140F, P	676	1957	159	197	1.0 (min)(3)
Viscosity, 212F, cSt	-	-	741	821	15.0 (min)(3)
Viscosity, 275F, cSt	254	417	-	-	—
after RTFO:					
Viscosity, 140F, P	1532	4333	333	377	—
Weight Loss, %	-	-	0.77	0.47	1 0 (max)(3)
Viscosity Ratio,	2.27	2.21	2.09	1.91	3 0 (max)(3)(13)
Composition:					
Saturates, %	15.20	13.03	18.38	17.43	—
Asphaltenes, %	23.45	25.25	20.25	20.65	—
Aromatics, %	23.11	20.27	24.68	23.99	—
Polars, %	38.24	41.45	36.69	37.93	—
Polars ÷ Saturates	2.52	3.18	2.00	2.18	0.5(min)(→)
Asphaltenes+Saturates	38.65	38.28	38.63	38.08	40(max)(App.A)
Solubility:					
Asphaltene Peptizability, (P_a)	0.68	0.67	0.70	0.68	—
Maltene Peptizing Power, (P_o)	1.20	1.23	0.97	1.11	3.0±0.5(min)
State of Peptization, (P)	3.75	3.77	3.22	3.45	(App.A)
Limiting Dilution Ratio, (X_{min})	2.75	2.77	2.22	2.45	—
Limiting Titrant Volume, (T_o)	2.92	2.97	2.24	2.34	—
Waxman's Cotangent	2.06	1.98	2.32	2.15	—

Note 1: Viscosity at 39.2F and 77F by Schweyer Apparatus(9)
Viscosity at 100F and 140F by ASTM D2171
Viscosity at 212F and 275F by ASTM D2170

Note 2: MBD-D &
MBD-E are AC-10
& AC-20, resp.

46

However, based on the criteria stated before for Tyndall modifiers, the MBD-G modifier appears the best overall choice for use. Viscosity ratio and asphalteness + saturates are lowest, and tne state of peptization represents about the average of the four materials. According to the variability limits in Appendix A, the above four modifiers may be said to be equally preferable on a composition and solubility basis.

7.   Modifier/RAP Binder Blends

The four modifiers were each blended with extracted and recovered asphalt from RAP taken from pavements in Phase B of reconstruction. Each blend contained varying quantities of recycling agent, shown in Table 17, so that viscosity of the blend lies within viscosity tolerances specified at 140 °F in ASTM D 3381 for AC-40. Physical and chemical properties of these blends are shown in Tables 17 and 18. Blends are identified with the numeral 5 added to the modifier identification, as MBD-D5, for modifier MBD-D blended with RAP binder.

A comparison of blend properties may give an indication of which blended material will have the most desirable performance in the field. Comparing properties as for the Tyndall blends:

	MBD-D5	MBD-E5	MBD-F5	MBD-G5
• Slope of viscosity/temerature curve	9.75	9.73	9.87	9.94
• Weight loss, percent	-	-	-	-
• Viscosity ratio	2.25	2.31	2.28	2.04
• Asphaltenes + Saturates*	45.82	43.18	47.74	47.17
• State of peptization*	3.11	2.89	2.67	2.90
• Percent modifier	58	78	40	42

---

*After RTFO aging

47

TABLE 17. MACDILL MODIFIER/RAP BINDER PHYSICAL PROPERTIES

Property	Modifier/RAP Binder Blend				RAP Binder	AC-40 Spec ASTM D3381
	MBD-D5	MBD-E5	MBD-F5	MBD-G5		
before RTFO:						
Viscosity, 77F, P x E3	389	398	350	383	-	-
Viscosity, 140F, P	3446	3630	3679	3887	64721	3200-4800
Viscosity, 275F, cSt	478	509	468	474	1434	300 or 400min
Slope LogVis/LogTemp	9.75	9.73	9.87	9.94	12.46	-
Penetration, 77F, 0.01mm	49	45	38	37	17	20 or 40 min
Penetration, 39.2F, 0.01mm	15	8	12	12	4	-
Ductility, 60F, cm	100+	100+	40	38	-	-
after RTFO:						
Viscosity, 140F, P	7750	8395	8398	7912	105000	-
Viscosity Ratio,	2.25	2.31	2.28	2.04	1.62	-
Penetration, 77F, 0.01mm	30	30	39	30	16	-
Penetration, 39.2F, 0.01mm	5	3	16	18	8	-
Penetration Retained, %	61	67	80	81	94	-
Ductility, 60F, cm	15	16	12	16	-	-
Modifier, % by blend weight required for 4000P viscosity	58	78	40	42	-	-

Note: Viscosity at 140F by ASTM D2171
Viscosity at 275F by ASTM D2170
Viscosity at 77F by Schweyer Rheometer (9)

TABLE 18. MACDILL MODIFIER/RAP BINDER BLEND, CHEMICAL PROPERTIES

| | Modifier/RAP Binder Blend | | | | | | | | | RAP | |
| | MBD-D5 | | MBD-E5 | | MBD-F5 | | MBD-G5 | | | | |
Property	Before RTFO	After RTFO	Before RTFO	After RTFO	Before RTFO	After RTFO	Before RTFO	After RTFO	Before RTFO	After RTFO
Composition:										
Saturates, %	15.34	15.38	14.00	13.42	16.89	16.98	16.51	16.65	15.50	16.40
Asphaltenes, %	27.72	30.44	26.53	29.76	30.04	30.76	28.98	30.52	35.88	37.49
Aromatics, %	19.20	16.42	19.44	17.17	17.73	17.09	18.78	16.92	14.13	12.63
Polars, %	37.77	37.76	40.03	39.65	35.33	35.17	35.72	35.91	34.98	33.48
Polars + Saturates	2.46	2.46	2.86	2.95	2.09	2.07	2.16	2.16	2.26	2.04
Asphaltenes+Saturates	43.06	45.82	40.53	43.18	46.93	47.74	45.49	47.17	51.38	53.89
Solubility:										
Asphaltene Peptizability, (P_a)	0.52	0.58	0.66	0.67	0.58	0.64	0.53	0.61	0.59	0.59
Maltene Peptizing Power, (P_o)	1.59	1.32	1.14	0.96	1.23	0.95	1.41	1.14	1.03	1.17
State of Peptization, (P)	3.27	3.11	3.37	2.89	2.93	2.67	3.00	2.90	2.48	2.84
Limiting Dilution Ratio, (X_{min})	2.27	2.11	2.37	1.89	1.93	1.67	2.01	1.90	1.48	1.85
Limiting Titrant Volume, (T_o)	2.47	1.96	2.23	2.01	2.11	1.44	2.04	2.04	1.68	2.01
Waxman's Cotangent	0.99	1.41	2.03	1.94	1.29	1.92	1.12	1.48	1.32	1.35

49

After modification, the MacDill blends display somewhat more varied properties than the Tyndall blends. The difficulty in choosing the most desirable modifier lies in determining whether the differences seen between parameters are significant. In other words, for state of peptization, is 2.67 significantly different than 3.11? If so, for asphaltenes + saturates, is 45.82 significantly different than 47.74? If these values for these parameters can be shown as significant, the next question involves absolute value of each parameter. It is known that low asphaltenes + saturates and high state of peptization are desirable, but, given a conflict between materials, as in this case, which should be chosen? The outline below may be of some help.

Parameter	Desire	Material Which Satisfies
• Slope of viscosity/ temperature curve	↓	MBD-E5
• Viscosity ratio	↓	MBD-G5
• Asphaltenes + Saturates*	↓	MBD-E5
• State of peptization*	↑	MBD-D5
• Percent Modifier	↓	MBD-F5

Deciding upon a modifier is difficult, since all four benefit the RAP after modification in some way. MBD-E satisfies more parameters than the others, but are the parameters it satisfies as significant to pavement performance as the remaining three parameters? If cost is no object, modifier MBD-F can be eliminated because it satisfies no performance parameters.

Answers to these questions are not available at this writing because no field performance data exist which could substantiate claims as to which parameter or combination is the most important.

---

* After RTFO aging

C. HURLBURT FIELD

1. RAC Cores

Test results from seven cores obtained at Hurlburt and provided to
NMERI for testing are summarized in Table 19. Test results of 10 from 17
additional cores evaluated by the paving contractor appear in Table 20. The
NMERI-evaluated core data in Table 19 indicate a substantial variation in
binder content and air voids. Binder content variation ranges from 4.7 to 6.6
percent for Cores 13 and 23 and Core 17, respectively. Air voids variation
ranges from 1.2 percent for core 17 to 6.9 percent for Core 25. Table 19
indicates the range for the 95 percent confidence limits for the mean of each
parameter and identifies values out of this range. Average Marshall stability
for the seven cores is 1030 pounds with standard deviation of 310 pounds. One
core had a stability of 1665 pounds, well outside the upper 95 percent
confidence limits for mean stability. This same core is also well above the
95 percent confidence limits for resilient modulus before and after water
treatment. The anomoly is that this core has next to the highest binder
content and next to the lowest air voids. The stability data in Table 19 are
presented for general information and not to invalidate the contractor's data
in Table 21.

Data measured by the paving contractor on 10 additional core discrepancy
samples compare core density with laboratory-compacted density of loose mix
representative of in-place materials. These data are presented in Table 20
and show the relative compaction of the in-place mix. Table 20 shows 95
percent confidence limits for the mean core and laboratory densities.
Compaction percent was not calculated for core or laboratory specimens lying
outside the 95 percent confidence limits for either density parameter.

2. Loose RAC

Data presented in Table 21 are densities and Marshall properties for
loose RAC compacted in the laboratory and tested by the paving contractor.
Notice that average Marshall stability is considerably higher than shown in
Table 19; however, specific gravity and flow are approximately equal. This
discrepancy in stabilities supports the claim that field and laboratory
Marshalls are not comparable.

TABLE 19. RAC CORE PROPERTIES, NMERI EVALUATION

Core No.	Average Binder, %	Thickness, in	Bulk Specific Gravity	Air Voids, %	Marshall Stability, lbs	Marshall Flow, .01 in	Resilient Modulus Dry, psi ×1000	Wet, psi ×1000
1	6.5	2.05	2.332	3.1	1665	16	370	510
6	5.2	1.95	2.331	5.7	860	15	165	250
13	4.7	2.18	2.313	4.1	900	15	155	200
17	6.6	2.09	2.372	1.2	1250	13	230	280
20	6.4	2.08	2.310	4.4	700	13	130	260
23	4.7	2.10	2.341	4.3	1070	14	155	200
25	5.1	2.07	2.307	6.9	770	13	165	280
95% Confid Interval	4.8 to 6.4	2.01 to 2.14	2.308 to 2.350	2.5 to 5.9	720 to 1340	13 to 15	120 to 275	180 to 380

52

TABLE 20. RAC CORE PROPERTIES, CONTRACTOR EVALUATION

Core No.	Core Specific Gravity	Loose Mix Specific Gravity	Compaction, %
1E	2.323	2.349 *	
1W	2.349	2.347 *	
2E	2.338	2.346 *	
2W	2.325	2.356	98.7
3E	2.321	2.356	98.5
3W	2.319	2.362 *	
4E	2.338	2.362 *	
4W	2.323	2.368 *	
5E	2.135 *	2.355	
5W	2.313	2.355	98.2
95% Confid Limits	2.264 to 2.352	2.351 to 2.361	98.3 to 98.7

TABLE 21. LABORATORY-COMPACTED LOOSE RAC, CONTRACTOR EVALUATION

Core No.	Specific Gravity	Marshall Stability, lbs	Marshall Flow, .01 in
1	2.365	2565	16
2	2.349	2285	16
3	2.346	2510	16
4	2.356	2220	15
5	2.362	2580	14
6	2.368	2410	15
7	2.355	2540	14
95% Confid Interval	2.349 to 2.365	2310 to 2570	14 to 16

3. RAC Core Binder

Physical and chemical properties of binders obtained from the seven cores submitted to NMERI for testing appear in Table 22. Recovered binder viscosity varies between cores, as might be expected. Core 1 has the highest viscosity and correspondingly lowest penetration. Since locations of cores are unknown, speculation for this difference cannot be made. However, Core 1 also had the highest stability and resilient modulus. This comparison simply suggests that a wide variation in material properties occurs within the recycled pavement.

4. RAC Binder From Laboratory Compacted Specimens

Physical and chemical properties of binder from laboratory-molded RAC appear in Table 23. Note that viscosity at 140 °F and penetration at 77 °F match values obtained for Core 1 from Table 22. This suggests that a wide variation in physical properties exists for Hurlburt recycled materials. However, the variation seen in physical properties is not present for chemical properties. This suggests that a poor correlation exists between viscosity and penetration and the composition and solubility parameters. Table 24 is a summary of average composition and solubility parameters for Table 22 and Table 23 materials. Core 1 was included in the average for one column in Table 24 and not included in another column. This was done to preclude any bias in the average results by this binder of significantly higher viscosity. Note the insignificant difference between Column 1 and Column 2 data, but an apparent difference between these and Column 3 data. Core 1 and other Column 1 cores have essentially equal chemical properties (little difference between Columns 1 and 2), but Columns 1 and 2 and Column 3 have dissimilar chemical properties. This comparison further suggests a poor correlation exists between physical and chemical properties for the recycled binders at Hurlburt. However, Column 3 recycled mixtures were reheated in the laboratory.

This lack of correlation between physical and chemical properties pinpoints the significance of conducting a thorough preliminary material analysis to establish the potential for variabilities in the existing pavement materials. Establishment of a material variability profile can lead to

TABLE 22. HURLBURT RAC CORE BINDER PHYSICAL AND CHEMICAL PROPERTIES

Property	Core Binder						
	1	6	13	17	20	23	25
Viscosity, 140F, P	34842	7491	2643	6095	2896	1032	5931
Penetration, 77F, .01 mm	24	41	63	41	-	123	47
Penetration, 39.2F, .01 mm	8	17	27	20	-	57	22
Composition:							
Saturates, %	17.51	18.15	22.37	21.86	17.47	16.53	18.78
Asphaltenes, %	33.09	29.04	25.08	26.94	28.77	26.51	28.87
Aromatics, %	14.43	14.61	12.75	12.84	14.48	14.89	13.89
Polars, %	34.96	38.20	39.80	38.36	39.29	42.06	38.46
Polars ÷ Saturates	2.00	2.10	1.78	1.75	2.25	2.54	2.05
Asphaltenes+Saturates	50.60	47.19	47.45	48.80	46.24	43.04	47.65
Solubility:							
Asphaltene Peptizability,(P_a)	0.55	0.55	0.63	0.60	0.54	0.57	0.51
Maltene Peptizing Power,(P_o)	1.06	1.16	1.13	1.28	1.08	1.01	1.39
State of Peptization, (P)	2.38	2.57	3.03	3.20	2.33	2.30	2.95
Limiting Dilution Ratio,(X_{min})	1.38	1.57	2.03	2.20	1.33	1.30	1.85
Limiting Titrant Volume,(T_o)	1.43	1.66	2.22	2.19	1.45	1.41	1.88
Waxman's Cotangent	1.22	1.18	1.60	1.51	1.08	1.24	1.04

TABLE 23. HURLBURT RAC LABORATORY-COMPACTED PHYSICAL AND CHEMICAL PROPERTIES

Property	Core Binder			
	1	2	3	4
Viscosity, 39.2F, x E06 P	856	768	567	475
Viscosity, 77F, x E06 P	2.0	2.2	1.8	1.8
Viscosity, 140F, P	24985	30372	25055	27643
Viscosity, 275F, cSt	901	1076	821	976
Penetration, 77F, .01 mm	26	24	27	26
Penetration, 39.2F, .01 mm	14	13	15	13
Ductility, 60F, cm	5	5	5	5
Composition:				
Saturates, %	15.5	15.3	15.8	15.8
Asphaltenes, %	34.6	34.8	34.0	34.6
Aromatics, %	16.1	15.8	16.0	15.5
Polars, %	33.8	34.2	34.4	34.0
Polars ÷ Saturates	2.2	2.2	2.2	2.2
Asphaltenes+Saturates	50.1	50.1	49.8	50.4
Solubility:				
Asphaltene Peptizability, (P_a)	0.51	0.51	0.52	0.52
Maltene Peptizing Power, (P_o)	1.08	1.08	1.05	1.03
State of Peptization, (P)	2.21	2.22	2.18	2.13
Limiting Dilution Ratio, (X_{min})	1.21	1.22	1.18	1.13
Limiting Titrant Volume, (T_o)	1.27	1.32	1.27	1.24
Waxman's Cotangent	1.03	1.01	1.03	1.02

TABLE 24.   HURLBURT BINDER CHEMICAL PROPERTIES COMPARED, CORES VERSUS
LABORATORY-COMPACTED

Composition:	Table 22 Average w/ Core No. 1	Table 22 Average w/o Core No. 1	Table 23 Average Table 23
Saturates, %	18.95	19.19	15.60
Asphaltenes, %	28.33	27.54	34.50
Aromatics, %	13.98	13.91	15.85
Polars, %	38.73	39.36	34.10
Polars ÷ Saturates	2.07	2.08	2.19
Asphaltenes+Saturates	47.28	46.73	50.10

Solubility :			
Asphaltene Peptizability,(P_a)	0.56	0.57	0.52
Maltene Peptizing Power,(P_o)	1.16	1.18	1.06
State of Peptization, (P)	2.68	2.73	2.19
Limiting Dilution Ratio,(X_{min})	1.67	1.71	1.19
Limiting Titrant Volume,(T_o)	1.75	1.80	1.28
Waxman's Cotangent	1.27	1.28	1.02

selection of one or a multiple of modifier products to meet the prevailing needs. This approach can assist in closing the gap of disagrement as observed in this part of the study.

5. RAP Binder

Physical and chemical properties of RAP binder are presented in Table 25. Extractions of RAP yielded binder content as shown in the table.

6. Modifiers

Results of physical and chemical laboratory tests on the two modifiers evaluated from Hurlburt Field are shown in Table 26. Certain recommended limiting values (Reference 4 and Appendix A) are also shown for some of these test parameters to help identify which of the modifiers tested appears the most desirable for recycling purposes.

Characteristics of modifiers discussed earlier for Tyndall and MacDill materials which are also important to the recycled pavement at Hurlburt are shown below.

	MBD-2D	MBD-2DD
• Slope of viscosity/temperature curve	9.09	9.12
• Weight loss, percent	0.03	0.63
• Viscosity ratio	2.13	2.07
• Asphaltenes + Saturates	40.93	42.77
• State of peptization (Before RTFO)	3.06	2.90
(After RTFO)	2.63	2.77

Based on the criteria stated before for Tyndall modifiers, either modifier appears adequate for use as a recycling agent. Temperature susceptibility, asphaltenes + saturates, and weight loss are lowest, and state of peptization highest, before RTFO for MBD-2D. However, after RTFO, MBD-2DD has a higher state of peptization. MBD-2D hardens somewhat more than MBD-2DD, but overall, properties are similar.

59

TABLE 25.   HURLBURT RAP BINDER PHYSICAL AND CHEMICAL PROPERTIES

| Property | Run | | | |
	1	2	3	Avg.
Viscosity, 140F, P	310024	331458	-	320741
Binder, %	6.64	6.58	6.68	6.63
Composition:				
Saturates, %				20.95
Asphaltenes, %				32.23
Aromatics, %				10.20
Polars, %				36.62
Polars ÷ Saturates				1.75
Asphaltenes+Saturates				53.18
Solubility:				
Asphaltene Peptizability, (P_a)				0.60
Maltene Peptizing Power, (P_o)				1.04
State of Peptization, (P)				2.64
Limiting Dilution Ratio, (X_{min})				1.64
Limiting Titrant Volume, (T_o)				1.53
Waxman's Cotangent				1.58

TABLE 26. HURLBURT MODIFIER PHYSICAL AND CHEMICAL PROPERTIES

Property	MBD-2D	MBD-2DD	Limits, Ref in ()
Before RTFOT:			
Viscosity, 100F, P	8133	9719	
Viscosity, 140F, P	285	310	>1 (3)
Viscosity, 212F, cSt	1005	1174	>15 (3)
Flash Point, F	615	395 *	>450 (3)
After RTFOT:			
Viscosity, 140F, P	607	643	
Weight Loss, %	0.03	0.63	<1.0 (3)
Viscosity Ratio	2.13	2.07	<3.0 (3)
Composition, before RTFOT:			
Saturates, %	20.94	24.20	-
Asphaltenes, %	19.99	18.57	-
Aromatics, %	22.82	19.02	-
Polars, %	36.26	38.22	-
Polars ÷ Saturates	1.73	1.58	>0.5 (3)
Asphaltenes+Saturates	40.93	42.77	>40 (App.A)
Solubility, before RTFOT:			
Asphaltene Peptizability, (P_a)	0.64	0.63	-
Maltene Peptizing Power, (P_o)	1.09	1.06	-
State of Peptization, (P)	3.06	2.90	>3.0±0.5
Limiting Dilution Ratio, (X_{min})	2.06	1.90	-
Limiting Titrant Volume, (T_o)	1.87	1.83	-
Waxman's Cotangent	1.90	1.77	-
Solubility, after RTFOT:			
Asphaltene Peptizability, (P_a)	0.61	0.61	-
Maltene Peptizing Power, (P_o)	1.03	1.08	-
State of Peptization, (P)	2.63	2.77	>3.0±0.5
Limiting Dilution Ratio, (X_{min})	1.63	1.77	-
Limiting Titrant Volume, (T_o)	1.61	1.85	-
Waxman's Cotangent	1.56	1.51	-

*

Test discontinued due to foaming and splattering.

7. Modifier/RAP Binder Blends

The two recycling agents were each blended with extracted and recovered asphalt from RAP. Each blend contained 66 percent recycling agent by weight so that viscosity of the blend lies within 2000 ± 400 poises viscosity at 140 °F. Physical and chemical properties of these blends are shown in Tables 27 and 28. Blends are identified with the letter H added to the modifier identification, as MBD-2DH, for modifier MBD-2D blended with RAP binder.

A comparison of blend properties may indicate which blended material will have the most desirable performance in the field. Comparing properties as for the Tyndall and MacDill blends:

	MBD-2DH	MBD-2DDH
• Slope of viscosity/temperature curve	9.28	9.41
• Weight loss, percent	-	-
• Viscosity ratio	2.00	2.36
• Asphaltenes + Saturates*	45.35	45.49
• State of peptization*	2.77	2.64

Based on the data shown, either modified system would be sufficient and compatibility between either modifier and RAP binder can be considered equivalent.

8. RAC Aggregates

RAC Cores were extracted and the gradation of aggregates obtained. Results of these tests appear in Table 29. Aggregates from the 14 cores evaluated by the paving contractor also appear for comparison with job mix formula and specification grading. Note that while exact comparison between contractor data and NMERI data is not present, a very close relationship exists. Also, average grading of the NMERI core aggregates and contractor aggregates meet the specification limits.

---

* After RTFO aging

62

TABLE 27. HURLBURT MODIFIER/CORE BINDER PHYSICAL PROPERTIES

Property	Blend	
	MBD-2DH	MBD-2DDH
before RTFO:		
Viscosity, 77F, P x E6	7.4	9.5
Viscosity, 140F, P	1685	1924
Viscosity, 275F, cSt	320	334
Slope LogVis/LogTemp	9.28	9.41
Penetration, 77F, 0.01mm	75	68
Penetration, 39.2F, 0.01mm	29	28
Ductility, 60F, cm	88	91
after RTFO:		
Viscosity, 140F, P	3378	4545
Viscosity Ratio,	2.00	2.36
Penetration, 77F	50	42
Penetration Retained, %	67	62
Ductility, 60F, cm	20	19

Note: Viscosity at 140F by ASTM D2171
      Viscosity at 275F by ASTM D2170
      Viscosity at 77F by Schweyer Rheometer (9)

TABLE 28. HURLBURT MODIFIER/CORE BINDER BLEND, CHEMICAL PROPERTIES

| Property | Modifier/Core Binder Blend | | | |
| | MBD-2DH | | MBD-2DDH | |
	Before RTFO	After RTFO	Before RTFO	After RTFO
Composition:				
Saturates, %	21.06	20.11	22.38	21.44
Asphaltenes, %	23.24	25.24	22.26	24.05
Aromatics, %	18.17	17.95	15.69	16.55
Polars, %	37.54	36.71	39.66	37.97
Polars ÷ Saturates	1.78	1.83	1.77	1.77
Asphaltenes+Saturates	44.30	45.35	44.64	45.49
Solubility:				
Asphaltene Peptizability, (P_a)	0.62	0.61	0.61	0.61
Maltene Peptizing Power, (P_o)	0.99	1.08	1.04	1.03
State of Peptization, (P)	2.62	2.77	2.63	2.64
Limiting Dilution Ratio, (X_{min})	1.62	1.77	1.63	1.64
Limiting Titrant Volume, (T_o)	1.67	1.85	1.61	1.53
Waxman's Cotangent	1.63	1.51	1.56	1.58

TABLE 29. AGGREGATE GRADATION, HURLBURT CORES

% Passing
Core No.

Sieve Size	1	6	13	17	20	23	25	Contractor Avg. Result	Job Mix Formula	Spec Range
3/4"	100	100	100	100	100	100	100	100	100	100
1/2"	97	100	97	95	98	97	98	97	97	96-100
3/8"	90	93	89	86	91	88	89	87	87	79-93
4	72	67	67	70	69	68	67	66	66	59-75
8	56	52	53	56	54	57	54	53	54	46-60
16	46	40	43	47	43	46	43	42	42	34-48
30	32	27	29	32	29	31	30	30	31	24-38
50	16	15	16	16	16	15	16	19	21	15-27
100	7	7	8	6	8	7	8	10	11	8-18
200	3.3	2.8	3.7	3	3.8	2.6	3.6	6.3	6.2	3-7

9.   RAP Aggregates

RAP obtained during construction was extracted from asphalt binder and aggregates sieved to produce the gradings shown in Table 30. Also shown in the table is the average result of sieve analyses performed by the paving contractor on a small-scale sample of RAP obtained prior to construction. Note close comparison on all sieves down to the Number 50 sieve. The finer contractor aggregates below the Number 50 sieve are apparently due to differences between small-scale and large-scale milling operations.

TABLE 30. HURLBURT RAP AGGREGATES

% Passing

Sieve Size	Test No. 1	Test No. 2	Test No. 3	Avg.	Contractor Prelim *
3/4"	100	100	100	100	100
1/2"	98	97	95	97	98
3/8"	96	94	90	93	92
4	86	80	78	81	82
8	73	66	67	69	74
16	63	58	58	60	58
30	49	48	43	47	46
50	26	23	20	23	33
100	12	9	9	10	19
200	6	5	4	5	11

*
Courtesy of Okaloosa Asphalt Enterprises, Inc.

67

SECTION V
CONCLUSIONS

A. TYNDALL AFB

1. Testing of cores from Tyndall Runway 13R-31L indicates density of cores, viscosity of binder from cores, and gradation of aggregate from cores to be relatively uniform along the runway length.

2. Three recycling agents were evaluated from Tyndall AFB. All three meet the "Tentative Recycling Agent Selection" criteria and all three came from a single source.

3. Recycling agent MBD-C appears to be the most desirable of the Tyndall recycling agents based on certain physical and chemical properties.

4. Asphalt properties were measured for compacted test specimens after aging in a 140 °F oven up to 123 days. Of the tests performed, conventional viscosity measurements appear to be the best means to measure changes in the aged asphalt. Viscosity and penetration may also be better predictors of bulk changes in asphalt behavior for asphalt aged in the rolling thin film oven.

B. MACDILL AFB

1. Asphalt concrete from Taxiway 7 (Phase A) was rather nonuniform after recycling based on test results from 14 cores taken at six different locations. Uniformity was judged based on density and thickness of cores, viscosity and penetration of binders from cores, and aggregate gradation.

2. Four recycling agents were used in Phase A reconstruction; one was used in Phase B recycling. All meet the "Tentative Recycling Agent Selection" criteria.

3. Prior to blending with RAP binder, modifier MBD-G appeared best suited for recycling. However, after blending, modifier MBD-D AND MBD-E appear to influence blend performance significantly better than any of the others. This is measured by the significantly higher 60 °F ductility before RTFU conditioning.

4. Physical tests of RAC core binders appeared more sensitive to changes in material types than chemical tests.

C.   HURLBURT FIELD

1. Modifiers used at Hurlburt met criteria for physical requirements. This did not meet chemical criteria for asphaltenes + saturates. Both modifiers exceeded the 40 percent limit by 1 percent and 3 percent, respectively, for MBD-2D and MBD-2DD. However, these deviations are within the variability limits presented in Appendix A.

2. A large difference was evident between physical properties of RAC cores and laboratory compacted RAC. The laboratory-compacted materials were generally stiffer, that is, had higher resilient moduli and Marshall stabilities with equivalent flow values. The difference in stiffness was probably due to the reheating process in preparing the laboratory-compacted specimens.

3. Physical and chemical properties of RAC recovered binder do not appear to correlate well. Where physical properties such as viscosity and penetration showed differences between various materials, chemical properties did not. And, when chemical properties suggested differences, physical properties did not.

D.   GENERAL CONCLUSIONS

The results of the effort have indicated that choices of a best-candidate modifier can be difficult based on a single set of properties. Also some of the results have shown that viscosity may be more sensitive to measure changes during aging conditions of binders than chemical properties. Discussions with many experts in asphalt chemistry indicate that chemical changes of even 10 percent can result in substantial changes in physical properties. Thus, what may appear to be a very significant change in physical properties numerically may likewise be a very significant change on a chemical basis.

Since the modifiers were not chosen using the criteria outlined in the tentative modifier selection specification, to make any inferences about performance would be inappropriate. To validate the selection criteria, the modifier selected should meet the specification before use.

The results in this study have not totally indicated the independent adequacy of physical tests from chemical tests in evaluating modifiers for hot recycling applications. Such proof can only be determined from a properly designed experiment in which variables can be controlled.

SECTION VI
RECOMMENDATIONS

1. Field testing is needed to verify test criteria for modifiers and blends of modifiers and RAP binder. Blending RAP binder and modifiers to equivalent viscosity does not provide equivalent physical or chemical properties. However, before criteria can be affirmed for other physical and chemical parameters, full-scale field tests are needed to help identify these other significant physical and chemical criteria and to help establish limits for specification purposes.

2. Existing design and construction criteria for RAC are essentially equivalent to those for virgin asphalt concrete. However, benefits of recycling agents in RAC mixtures have been observed. The incorporation of these benefits in mix designs is lacking and, therefore, more effort is required to establish the connection.

3. The blending criteria used in this research assume all RAP binder combines with the recycling agent to produce the target viscosity. Unfortunately, no procedure exists to determine if this assumption is accurate. Certainly, less than 100 percent of RAP binder actually combines. Therefore, the actual viscosity of RAC binder after recycling may be higher than designed. This may produce a stiffer mixture than planned, leading to potentially crack-susceptible recycled pavements.

4. Needed is a method to estimate the actual quantity of RAP binder which combines with recycling agents. This estimate could then be applied to mixture designs so that mixtures with appropriate binder viscosity are produced in the field.

71

REFERENCES

1. Smith, R. W., "A Summer of Recycling--An Update on Asphalt Pavement Recycling," **Paving Forum,** pp 5-9, 3.

2. Teh-Chang, L., Terrel, R. L., and Mahoney,J. P., "Measurement of Mixing Efficiency in Pavement Recycling," AAPT, Vol, 52, 1983.

3. Ruth, B. E., et al., "Asphalt Binder Hardening--Causes and Effects," Presented during AAPT Meeting, February 1985.

4. Kiggundu, B. M., Nusser-Humphrey, B. J., and Zallen, D. M., **Recycling Agent Selection and Tentative Specifications,** ESL-TR-84-47, Engineering and Services Laboratory, Air Force Engineering and Services Center, Tyndall Air Force Base, Florida, 1985.

5. Minutes of the Eighteenth Pacific Coast Conference on Asphalt Specifications, San Francisco, CA, May 17-18, 1983.

6. Heithaus, J. J., "Measurement and Significance of Asphaltene Peptization," American Chemical Society, New York, N. Y., September 1960.

7. Schweyer, H. E., et al., "A constant Stress Rheometer for Asphalt Cements," AAPT, Vol. 45, 1976, pp 53-72.

8. **The Asphalt Institute Manual Series,** No. 2., (MS-2), May 1984 Edition.

9. **Recycling Manual,** Chevron U.S.A., Inc., March 1982.

10. "Florida Method of Test for Resistance to Abrasion of Small Size Coarse Aggregate by Use of the Los Angeles Machine," Designation: FM 1-T 069.

11. **Annual Book of ASTM Standards,** Volume 04.03, 1985.

12. **Annual Book of ASTM Standards,** Volume 05.02, 1985.

13. **Standard Practice For Classifying Hot Mix Recycling Agents,** ASTM-D-455286.

APPENDIX A
TENTATIVE MODIFIER SELECTION CRITERIA

A. BACKGROUND

The initial effort from FY 82 through FY 84 involving the development of
a tentative recycling agent selection criteria were summarized in Reference
A1 in Table 34. The parameters defined in Reference A1 were proposed modi-
fier or recycling agent properties which are to be used to select a modifier
for hot recycling applications. The selected properties were based on:

- Viscosity at 140 °F (60 °C) and 212 °F (100 °C)
- Flashpoint COC, °F
- Weight loss (RTFO/TFO), percent
- Chemical composition properties
    - Compatibility ratio (Polars/Saturates)
    - Percent saturates plus asphaltenes

Successive efforts in FY85 involved the study of more field-aged RAP
materials from a variety of climates. The binders recovered from the RAP
materials were each blended with a maximum of nine modifiers. Each of the
nine blends was made to satisfy the same consistency level, in particular,
viscosity at 140 °F. Then, each blend was tested for physical and chemical
properties. The physical properties consisted of viscosity, penetration,
and ductility at various conditions of treatment and temperature. The
chemical properties were determined using the modified Clay-Gel and Heithaus
test procedures. These test methods are listed in Appendix B and
Appendix C.

The results from the extended study were used to propose modifications
to the tentative recycling agent or modifier selection criteria to be dis-
cussed in this Appendix. In addition, an interlaboratory evaluation program
was designed to establish variability limits for the parameters contained in
the proposed modifier selection criteria.

B. VARIABILITY LIMITS DEVELOPMENT

1. Interlaboratory Study

Four laboratories were involved i   ne interlaboratory study in which various asphalts were tested. The data generated by each laboratory were used individually to develop an intralaboratory variability statement. All the data were then assembled and analyzed to develop an interlaboratory variability statement. Two documents were used in the analysis of data and in preparation of the variability statements. These were ASTM C 802-80 Standard Practice For Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials, and C 670-84 Standard Practice for Preparing Precision Statements for Test Methods for Construction Materials.

Fifteen bituminous samples were tested by each laboratory, using the modified Clay-Gel and Heithaus test procedures. The materials consisted of three samples of each of the following:

- Blend prepared by NMERI

- Aged binder extracted by each participating laboratory from the same RAP

- A NMERI-provided modifier

- A blend prepared by each participating laboratory using the same materials, proportions, and conditions

- An extracted aged binder by NMERI from similar RAP as in the second item.

All 15 samples were labeled as: A1, A2, A3, B1, B2, B3, C, D, E, F, G, H, I, J, and K. However, all A materials were the same, all B materials were identical, C, D, E and F, G, and H, as well as I, J, and K were replicates. The sample labeling was intended to produce triplicate tests without the knowledge of the testing personnel. The only exception involved test samples prepared within the participating laboratory.

Attachment 1 at the end of this Appendix lists data compiled, analyses, and variability statements developed for the modified Clay-Gel and Heithaus test procedures.

2.   Variability Data Discussion

The variability data from the analyses of test results are listed in Tables A1 and A2. Table A1 lists the standard deviations (1S) for within and between laboratory for each of the five materials and for each composition fraction. Included in Table A1, for comparison purposes, are the precision limits calculated by using the guidelines given in ASTM D 2007 for materials whose polar compound percentages exceed 20. All the materials tested in this study contained polar compounds in excess of 35 percent.

Discussion in successive paragraphs refers to data in Table A1. When discussion switches to data in Table A2, it is made clear.

The within laboratory standard deviations (1S) for materials A and B were smaller across the board than the same values for materials C, D, and E. Materials A and B were recovered RAP binder and a blend prepared by each laboratory. The fact that the respective standard deviations are small suggests that the recovery and blend preparation procedures within each laboratory were equally controlled. The same observation is made with respect to the between laboratory 1S values for materials A and B.

For material C, which is a NMERI-recovered binder, the 1S values for both within and between laboratory for all fractions except the aromatics are nearly equal. There is no obvious explanation for the deviation in aromatic 1S values; however, the latter are still within the range of calculated values from the ASTM D 2007 procedure.

Material D, a NMERI-supplied modifier, and material E, a NMERI-prepared blend, display slightly larger 1S values than other materials. No obvious explanation for this result is seen at this time; however, the comparison with the calculated values from ASTM D 2007 is still favorable.

TABLE A1. SUMMARY OF STANDARD DEVIATIONS FOR COMPOSITION DATA

MATERIAL	INDEX	COMPOSITION FRACTIONS				ASTM D 2007-80			
		ASP	SAT	ARO	POL	ASP	SAT	ARO	POL
A	WITHIN	0.51	0.50	0.51	0.77	----	0.80	3.10	4.30
	BETWEEN	1.00	1.37	2.22	1.37	----	1.10	6.50	6.76
B	WITHIN	0.69	0.91	1.44	0.84	----	0.80	3.10	4.31
	BETWEEN	0.98	1.62	2.80	1.78	----	1.10	6.50	6.77
C	WITHIN	1.57	1.52	2.74	2.00	----	0.80	3.10	4.31
	BETWEEN	1.58	1.56	4.41	2.63	----	1.10	6.50	6.77
D	WITHIN	2.04	0.66	1.66	1.89	----	0.80	3.10	4.21
	BETWEEN	2.22	1.22	5.30	3.18	----	1.10	6.50	6.62
E	WITHIN	1.85	1.39	2.15	0.94	----	0.80	3.10	4.31
	BETWEEN	1.97	1.69	4.17	2.75	----	1.10	6.50	6.79

A = Proposor/contractor recovered RAP binder   ASP = ASPHALTENES

B = Proposer/contractor prepared blend   SAT = SATURATES

C = NMERI recovered RAP binder   ARO = AROMATICS

D = NMERI modifier (NMERI supplied)   POL = POLARS

E = NMERI prepared blend   STANDARD DEVIATIONS IN %

76

TABLE A2. SUMMARY OF STANDARD DEVIATIONS (1S) FOR SOLUBILITY DATA

MATERIAL	INDEX	P_a	P	P_o	X_{min}	T_o	Cot ϕ
				SOLUBILITY PARAMETERS			
A	WITHIN	0.02	0.16	0.06	0.16	0.13	0.14
	BETWEEN	0.06	0.32	0.12	0.32	0.39	0.28
B	WITHIN	0.02	0.16	0.09	0.16	0.28	0.09
	BETWEEN	0.05	0.32	0.14	0.32	0.74	0.16
C	WITHIN	0.03	0.21	0.06	0.21	0.29	0.12
	BETWEEN	0.05	0.28	0.06	0.28	0.38	0.23
D	WITHIN	0.01	0.19	0.07	0.19	0.34	0.08
	BETWEEN	0.02	0.17	0.07	0.17	0.37	0.13
E	WITHIN	0.05	0.21	0.10	0.21	0.20	0.23
	BETWEEN	0.05	0.30	0.08	0.30	0.39	0.29

P_a - ASPHALTENE PEPTIZABILITY   P - STATE OF PEPTIZATION

P_o - MALTENE PEPTITIZING POWER X_{min} - LIMITING DILUTION RATIO

T_o - LIMITING TITRANT VOLUME   Cot ϕ - WAXMAN'S COTANGENT ANGLE

The calculated limits, based on the guidelines in ASTM D 2007, are listed in Table A1 on the right. The calculated values are for saturates, aromatics, and polars fractions only. The limits on asphaltenes are not included because the original materials tested and from which guidelines were developed, contained less than 1 percent asphaltenes by weight. The differences between the calculated values and the variability limits may be due to the following:

* The aromatic fraction in D 2007 is obtained by numerical difference between a theoretical 100 percent recovery and the sum of saturates, polars, and asphaltenes. In the modified Clay-Gel procedure, the aromatic fraction is determined analytically.

* In ASTM D 2007 the polar fraction is eluted with a solvent mixture of benzene/acetone. In the modified Clay-Gel procedure the polar fraction is eluted with toluene/acetone and a final rinse with methylene chloride.

* In ASTM D 2007 the quantity of n-pentane used to separate the maltenes from the asphaltenes is less than 200 mL. In the modified Clay-Gel procedure the amount of n-pentane used at this step exceeds 1000 mL.

Although the solvent type may not play a significant role in the development of variability and/or precision statements, this factor should be kept in mind when making comparisons. The solvent effect was not part of this study.

Table A2 lists standard deviations (1S) for the solubility parameters determined in this study by the modified Heithaus method. The within laboratory 1S values for P_a for all materials tested ranged from 0.01 to 0.05 whereas for between laboratory the range is 0.02 to 0.06. The corresponding coefficients of variation are 1.5 to 7.5 percent for within and 2.5 to 11.0 percent for between laboratory. These results suggest that P_a, the ability of asphaltenes to be peptized, can be determined with confidence.

The within laboratory 1S values for P range from 0.16 to 2.21 whereas for between laboratory the values are 0.17 to 0.32. The coefficients of variation ranged from 6.0 to 8.0 percent for within and 5.4 to 12.1 percent for between laboratory. P has been used in the literature to relate solubility to pavement performance; thus, the low 1S values obtained in this study suggest that this parameter could be determined with confidence.

The 1S values for P_0 are also small. There is not a great difference between the within and the between laboratory values, which suggests that the method can be used by different testing organizations and comparisons made.

The 1S values for T_0 and X_{min} should be very close to one another because the two parameters represent the same threshold amount of nonpolar solvent required to destablize the asphalt system and induce flocculation. However, this was not the case in this study. It is not clear why the disparity appears.

The last parameter is cot $\emptyset$, which is an indicator of dispersibility. This parameter can range in value from zero to very large numbers. The larger the value of cot $\emptyset$, the higher the dispersive character of the material, and hence, the more desirable. The 1S values shown in Table A2 are reasonably small for a potentially large parameter. Thus, the cot $\emptyset$ parameter can also be determined with confidence.

Considering that there are no published variability limits for the solubility parameters determined by the modified Heithaus method, the values obtained in this effort are proposed to be first-order approximations. They may be used for acceptance of test results when using the tentative recycling agent selection specification until precision limits are established. Determining precision limits will require a thorough round robin test program as defined in ASTM D 1749.

79

In summary, Table A3 lists variability limits for Clay-Gel and Heithaus parameters extracted from Tables A1 and A2. The values in Table A3 are suggested limits which can be used for acceptance of test results generated by using either of the referenced test methods.

C. MODIFICATION OF THE TENTATIVE SPECIFICATION

1. Background

The Tentative Specification was first proposed and presented in ESL-TR-84-47 (Reference A1). This specification consisted of physical and chemical properties which were identified to be characteristic of a suitable modifier for hot recycling applications. The specification has the following attributes:

a. It is generic in that it is developed using test results on a broad range of modifiers compared to developing a specification on the basis of regional products. The products of reference in this study and this specification are petroleum-based.

b. The specification takes into account the physical and chemical properties of the modifiers.

c. This specification attempts to account for the respective sensitivity of each aged asphalt to the action of a modifier by proposing that the required polar/saturate ratio of a modifier should be greater than or equal to twice the asphaltene fraction of the recovered RAP binder. Along with this compatibility constraint is the requirement that a modifier should not contain asphaltenes plus saturates (A+S) in a percent exceeding 30. It was observed that some aged asphalt systems were very sensitive to the action of modifiers with a saturate content exceeding 30 percent. The sensitivity referred to in this discussion was measured by a viscosity aging index which is defined as:

$$\text{Viscosity aging index} = \frac{\text{viscosity of aged residue (RTFO)}}{\text{viscosity of unaged residue}}$$

TABLE A3. SUGGESTED VARIABILITY LIMITS FOR CLAY-GEL/HEITHAUS PARAMETERS

ITEM	INDEX	STANDARD DEVIATION		COEFFICIENT OF VARIATION	
		1S	D2S	%1S	%D2S
COMPOSITION					
ASP	single operator	0.98	2.77	3.9	10.9
	multiple operator	1.61	4.56	6.1	17.3
SAT	single operator	0.71	2.01	4.2	12.0
	multiple operator	1.56	4.34	8.7	24.6
ARO	single operator	1.16	3.28	6.2	17.4
	multiple operator	3.77	10.66	21.8	61.8
POL	single operator	0.93	2.62	2.5	7.1
	multiple operator	2.30	6.51	6.2	17.4
ASP+SAT	single operator	1.69	4.78	8.1	22.9
	multiple operator	3.17	8.90	14.8	41.9
SOLUBILITY					
P_a	single operator	0.03	0.08	3.1	8.7
	multiple operator	0.05	0.13	7.9	22.3
P	single operator	0.16	0.45	6.1	17.2
	multiple operator	0.25	0.79	10.1	28.6
P_o	single operator	0.07	0.19	6.2	17.6
	multiple operator	0.09	0.26	8.7	24.5
X_{min}	single operator	0.17	0.48	9.0	25.4
	multiple operator	0.28	0.79	15.8	44.8
T_o	single operator	0.21	0.59	13.7	38.8
	multiple operator	0.45	1.29	25.6	72.4
Cot ϕ	single operator	0.10	0.28	7.3	20.6
	multiple operator	0.21	0.60	13.7	38.8

Note: Abbreviated symbols have previously been defined.

The viscosities used in the index defined above are determined at 140 °F. Blends of RAP recovered binders and modifiers whose viscosity aging indexes exceeded 3.0 were considered to be sensitive.

d. This specification considers internal phase stability of binders and proposes applications of internal phase or solubility properties. The internal phase stability properties of an asphalt or modifier are measured by the Heithaus test method. The significant parameter identified and cited in the technical literature to relate to pavement performance is the state of peptization (P). The first phase of this specification did not contain numerical values of P; however, recent work has provided these. These numerical values are discussed subsequently.

2. Modifications

a. Provisions for use of more viscous products other than modifiers/recycling agents as far as the chemical portion of the specification was concerned have been added. Efforts involving soft asphalts and more viscous bituminous products in recycling studies have been conducted in the NMERI laboratory. The composition test results for the various modifying products used in the laboratory study are listed in Table A4. These results indicate that more viscous products with percent (A+S) less than or equal to 38.5+/-1.6, at the 95 percent confidence level, would be recommended for hot recycling applications. Thus, a maximum value of this varible was set at 40 percent. Determination of variability of the (A+S) variable was expected from the interlaboratory study.

b. Numerical values of compatibility were defined. The variables are the state of peptization (P) and the limiting Dilution Ratio (X_{min}). The state-of-peptization value of interest pertains to asphaltene containing modifiers because it can only be determined for bituminous products which flocculate when tested by the Heithaus test method. A relationship was attempted to relate P and the percent (A+S) as shown in Figure A1. This relationship suggested that at a value of percent (A+S) of 40, a corresponding value of P was 3.00. All the modifying products above this line had been

TABLE A4. THE - ASPHALTENES + SATURATES - CRITERIA

MODIFIER	30 % LIMIT			MODIFYING PRODUCT	40 % LIMIT		
	% (A + S)	VISCOSITY @ 140^0F, P	P/S		% (A + S)	VISCOSITY @ 140^0F, P	P/S
MBD-3	25.74	540.0	3.12	MBD-2	41.13	490.0	1.72
MBD-3B	22.21	58.0	2.18	MBD-2B	39.53	324.0	1.98
MBD-5	23.66	2.8	1.18	MBD-2D	40.93	285.0	1.73
MBD-6B	30.08	90.0	2.00	MBD-6A	39.01	300.0	2.23
MBD-7A	22.17	1.0	0.70	MBD-A	40.43	581.0	2.06
MBD-7C	25.41	1.1	0.71	MBD-B	36.53	276.0	1.74
MBD-8A	18.08	1.8	1.24	MBD-C	32.67	65.0	1.34
MBD-8C	16.05	1.0	1.18	MBD-D	38.65	676.0	2.27
MBD-9	6.56	2.3	4.51	MBD-E	38.28	1957.0	2.21
MBD-10	24.86	519	3.24	MBD-F	38.63	159.0	2.09
				MBD-G	38.08	197.0	1.91

% (A + S)   -   % (ASPHALTENES + SATURATES) BASED ON MODIFIED ASTM D 2007
     P/S   -   POLARS / SATURATES BASED ON MODIFIED ASTM D 2007

30% LIMIT

n = 9
$\bar{X}_{95\%}$ = 23.14 ± 3.23 (excluded)
     (26.4 max)

n = 10
$\bar{X}_{95\%}$ = 21.48 ± 4.70 (included)
     (26.2 max)

40% LIMIT

n = 11
$\bar{X}_{95\%}$ = 38.53 ± 1.59

40 % max was proposed

83
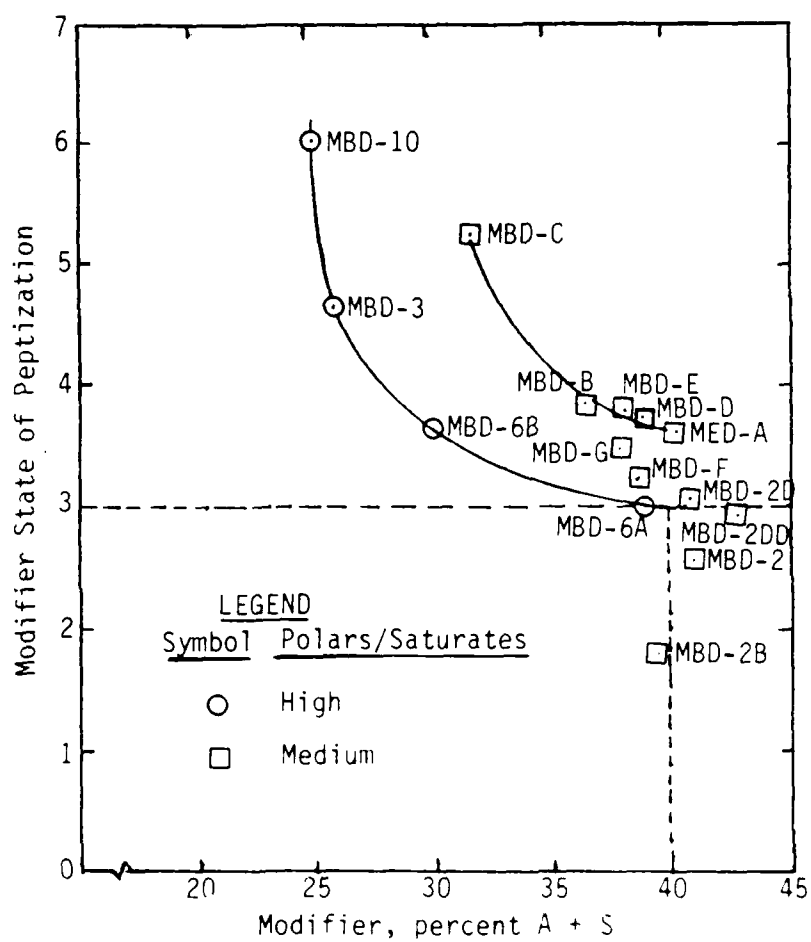
Figure A1. Modifier State of Peptization versus Percent
Asphaltenes + Saturates.

found to be acceptable for reconstituting aged binders. Further evidence about the value of P of 3.00 was presented in Reference A1 in a relationship developed to show the observed variation of viscosity aging index of a blend and the state of peptization of a modifier. Reference A1, showed that blend aging index of less than 3.00 was obtained with modifiers whose P value was greater than or equal to 3.00. Published results (Reference A2) were obtained recently and analyzed by NMERI in the form of viscosity aging index versus P. The P in the referenced report was determined using toluene and n-heptane. The analysis indicated that binders with viscosity aging indexes lower than 3.00 had P-values greater than or equal to 3. This study was concerned with classifying tender and nontender mixes for which virgin binders were used.

Most of the nontender mixes in the study under discussion were observed to occur in mixtures whose binder aging indexes were lower than 3.00. This observation suggests that binders with a P-value greater than or equal to 3.00 may produce nontender mixes. The variability limits about P determined from an interlaboratory study are listed in Table A3.

Because recycling agents used in hot recycling operations contain no asphaltenes, and hence a P-value cannot be established, there was a need to include coverage of the use of such products in the proposed specification. The results of the recycling agent selection study summarized in Reference A1 and subsequent efforts have overwhelmingly suggested that beneficial aspects of a recycling agent can be specified. The specification is that the X_{min} value of a blend after RTFO conditioning should be greater than the X_{min} value of the recovered RAP binder. An illustration of this concept is presented in Figure A2. The larger the shift or difference between these two X_{min} values the greater is the dispersive action of the modifier to the particular aged binder. However, should the reverse situation occur, the recycling agent/modifier is considered to be incompatible with the aged binder and an alternate modifier should be sought.

MATERIAL PROPERTIES

Material	Viscosity 140°F, Poises	D2007/ Heithaus Tests	Description
Control Asphalt	184,749	%Asphaltenes = 33.58 Heithaus, P = 2.61	Binder extracted from combined Runway/Taxiway RAP from Hurlburt Field
Modifier	519	Polar/Saturate = 3.24 Heithaus P = 6.03	AR - 1000
Blend MBD-108	2025 (unaged) 3803 (RTFO)	%Asphaltenes = 21.00 Heithaus P = 3.70 (RTFO Condition)	Blend target viscosity at 140°F, = 2000±400 Poises
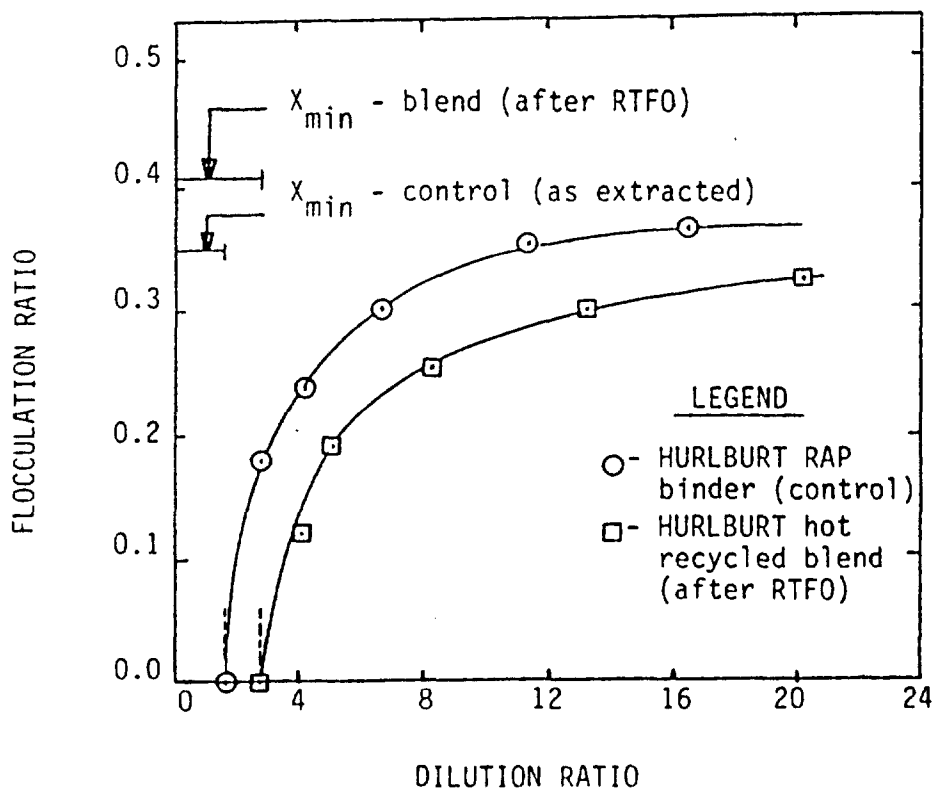
Figure A2. Illustrative Example of Expected Attributes of a Modifier.

3.   Tentative Specification

Table A5 lists the complete tentative criteria for modifier selection. The final blend made in accordance with the requirements in Table A5 MUST satisfy pertinent standard or local specifications for binders.

REFERENCES

A1. Kiggundu, B. M., Nusser-Humprey, B., and Zallen, D. M., **Recycling Agent Selection and Tentative Specification**, ESL-TR-84-47, Engineering and Services Laboratory, Air Force Engineering and Services Center, Tyndall Air Force Base, Florida, March 1985.

A2. Robertson, R. E., Ensley, E. K., and Petersen, J. C., **Physico-Chemical Studies of Tender Mix and non-Tender Mix Asphalts**, FHWA/RD-80/130, Department of Transportation, Federal Highway Administration, Washington, DC, December, 1980.

TABLE A5.   REQUIREMENTS FOR RECYCLING AGENTS/MODIFIERS FOR
            HOT CENTRAL PLANT RECYCLING OF ASPHALT PAVEMENTS

TEST PROPERTY		VALUE		REFERENCE
I.	**PHYSICAL**			
	1. Viscosity, 60 °C (140 °F), Poises	1.00	min	D 2171
	2. Viscosity, 100 °C (212 °F), cSt	15.00	min	D 2170
	3. COC Flash Point, °C	232	min	D 92
	4 Weight Loss (RTFO) at 325 °F for 85 minutes, %	1.00	max	D 2872
II.	**CHEMICAL**			
	A. Composition Analysis			D 2007 modified
	1. percent saturates + asphaltenes (recycling agents)	30	max.	
	2. percent saturates + asphaltenes (soft or harder asphalt grades)	40	max	
	3. Polar/Saturate Ratio	0.50 min or greater than or equal to twice the asphaltene fraction of the RAP binder.		

1

B. COMPATIBILITY (SOLUBILITY)

1. modifier state of peptization
    (P)   greater than
                                           3.00
2. state of peptization (P) for P_{min} blend RTFO
    blends made with recycling   should be greater
    agents after RTFO conditioning   than P_{min} of extracted
                                           RAP binder. see
                                           Figure A2.

Note 1. The reference solvent system for the compatibility limits
consists of toluene (polar) and n-dodecane (non-polar). The use of
other solvents may lead to other limits.

APPENDIX A

ATTACHMENT 1

DATA COMPILATION, ANALYSES, AND VARIABILITY STATEMENTS
DEVELOPED FOR MODIFIED GLAY-GEL AND HEITHAUS TEST PROCEDURES

VARIABILITY STATEMENT FOR CLAY-GEL COMPOSITIONAL ANALYSIS

The single-operator standard deviation has been found to be:

    0.98 % for asphaltenes
    0.71 % for saturates
    1.16 % for aromatics, and
    0.03 % for polars.

Therefore, results by the same operator on the same material should not differ by more than:

    2.77 % for asphaltenes
    2.01 % for saturates
    3.28 % for aromatics, and
    2.62 % for polars.

The multilaboratory standard deviation has been found to be :

    1.61 % for asphaltenes
    1.53 % for saturates
    3.77 % for aromatics, and
    2.30 % for polars.

Therefore, results of two properly conducted tests from two different laboratories on samples of the same material should not differ by more than:

    4.56 % for asphaltenes
    4.34 % for saturates
    10.66 % for aromatics, and
    6.51 % for polars.

The single-operator coefficient of variation has been found to be:

3.85% for asphaltenes
4.23% for saturates
6.15% for aromatics, and
2.51% for polars

Therefore, results for two properly conducted tests by the same operator on the same sample shold not differ from each other by more than:

10.90% for asphaltenes
11.97% for saturates
17.40% for aromatics, and
7.10% for polars

of their averages.

The multilaboratory coefficient of variation has been found to be:

6.11% for asphaltenes
8.69% for saturates
21.83% for aromatics, and
6.15% for polars

Therefore, results of two different laboratories on samples of the same material should no differ from each other by more than:

17.29% for asphaltenes
24.60% for saturates
61.77% for aromatics, and
17.39% for polars

of their averages.

STANDARD DEVIATIONS--1S, D2S,--FOR INTRA AND INTER LABS----CLAY-GEL

		1S	D2S	1S	D2S
		INTRA		INTER	
ASPHALTENES	PS	1.12	3.17		
	CTL	0.30	0.85	1.61	4.56
	WES	2.13	6.03		
	NME	0.36	1.02		
AVERAGE		0.98	2.77		
SATURATES	PS	0.62	1.75		
	CTL	0.16	0.45	1.53	4.34
	WES	1.80	5.09		
	NME	0.27	0.76		
AVERAGE		0.71	2.01		
AROMATICS	PS	1.36	3.85		
	CTL	0.32	0.91	3.77	10.66
	WES	2.57	7.27		
	NME	0.37	1.05		
AVERAGE		1.16	3.27		
POLARS	PS	1.05	2.97		
	CTL	0.43	1.22	2.30	6.51
	WES	1.79	5.07		
	NME	0.43	1.22		
AVERAGE		0.93	2.63		

COEFFICIENTS OF VARIATIONS--1S%, D2S%,--FOR INTRA AND INTER LABS---CLAY-GEL

		1S% INTRA	D2S%	1S% INTER	D2S%
ASPHALTENES	PS	4.31	12.19		
	CTL	1.10	3.11	6.11	17.29
	WES	8.74	24.73		
	NME	1.26	3.57		
AVERAGE		3.85	10.90		
SATURATES	PS	3.75	10.61		
	CTL	0.88	2.49	8.69	24.60
	WES	10.80	30.56		
	NME	1.47	4.16		
AVERAGE		4.23	11.97		
AROMATICS	PS	7.60	21.51		
	CTL	2.10	5.94	21.83	61.77
	WES	12.00	33.96		
	NME	2.91	8.24		
AVERAGE		6.15	17.40		
POLARS	PS	2.72	7.70		
	CTL	1.16	3.28	6.15	17.39
	WES	5.20	14.72		
	NME	0.96	2.72		
AVERAGE		2.51	7.10		

VARIABILITY STATEMENT FOR HEITHAUS COMPATIBILITY TEST

The single-operator standard deviation has been found to be:

0.17 for X_{min}

0.03 for P_a

0.16 for P

0.07 for P_o

0.21 for T_o

0.10 for cot 0.

Therefore, results by the same operator on the same material should not differ by more than:

0.48 for X_{min}

0.08 for P_a

0.45 for P

0.19 for P_o

0.59 for T_o, and

0.28 for cot 0.

The multilaboratory standard deviation has been found to be:

0.28 for X_{min}

0.05 for P_a

0.28 for P

0.09 for P_o

0.45 for T_o, and

0.21 for cot 0.

Therefore, results of two properly conducted tests from two different laboratories on samples of the same material should not differ by more than:

0.79 for X_{min}

0.13 for P_a

0.79 for P

0.26 for P_o

1.26 for T_o, and

0.60 for cot 0.

The single-operator coefficient of variation has been found to be:

    8.99% for Xmin
    3.06% for Pa
    6.08% for P
    6.21% for Po
  13.72% for To, and
    7.29% for Cot ϕ

Therefore, results for two properly conducted tests by the same operator on the same sample should not differ from each other by more than:

  25.44% for Xmin
    8.66% for Pa
  17.21% for P
  17.57% for Po
  38.83% for To, and
  20.63% for Cot ϕ

of their average.

The multilaboratory coefficient of variation has been found to be:

  15.83% for Xmin
    7.87% for Pa
  10.10% for P
    8.65% for Po
  25.57% for To, and
  13.69% for Cot ϕ

Therefore, results of two different laboratories on samples of the same material should not differ from each other by more than:

  44.81% for Xmin
  22.27% for Pa
  28.58% for P
  24.47% for Po
  72.37% for To, and
  38.75% for Cot ϕ

of their average.

STANDARD DEVIATIONS--1S, D2S,-- FOR INTRA AND INTER LABS----HEITHAUS

		1S	D2S	1S	D2S
		INTRA		INTER	
Xmin	PS	0.21	0.59		
	CTL	0.14	0.40	0.28	0.79
	WES	0.24	0.68		
	NME	0.07	0.20		
AVERAGE		0.17	0.47		
Pa	PS	0.05	0.14		
	CTL	0.006	0.017	0.05	0.13
	WES	0.05	0.14		
	NME	0.002	0.006		
AVERAGE		0.03	0.08		
P	PS	0.21	0.59		
	CTL	0.10	0.28	0.28	0.79
	WES	0.24	0.68		
	NME	0.08	0.23		
AVERAGE		0.16	0.45		
Po	PS	0.06	0.17		
	CTL	0.05	0.14	0.09	0.26
	WES	0.12	0.34		
	NME	0.04	0.11		
AVERAGE		0.07	0.19		
To	PS	0.38	1.08		
	CTL	0.12	0.34	0.45	1.29
	WES	0.25	0.71		
	NME	0.07	0.20		
AVERAGE		0.21	0.58		
COT ϕ	PS	0.20	0.57		
	CTL	0.04	0.11	0.21	0.60
	WES	0.13	0.37		
	NME	0.02	0.06		
AVERAGE		0.10	0.28		

COEFFICIENTS OF VARIATION--1S%, D2S%,--INTRA AND INTER LAB---HEITHAUS

		1S%	D2S%	1S%	D2S%
		INTRA		INTER	
Xmin	PS	14.45	40.89		
	CTL	5.80	16.41	15.83	44.81
	WES	11.79	33.37		
	NME	3.92	11.09		
AVERAGE		8.99	25.44		
Pa	PS	8.34	23.60		
	CTL	1.03	2.91	7.87	22.27
	WES	2.30	6.51		
	NME	0.55	1.56		
AVERAGE		3.06	8.66		
P	PS	8.54	24.17		
	CTL	3.63	10.27	10.10	28.58
	WES	9.54	27.00		
	NME	2.59	7.33		
AVERAGE		6.08	17.21		
Po	PS	5.09	14.40		
	CTL	4.91	13.90	8.65	24.47
	WES	11.50	32.55		
	NME	3.34	9.45		
AVERAGE		6.21	17.57		
To	PS	34.36	97.24		
	CTL	6.16	17.43	25.57	72.37
	WES	10.93	30.93		
	NME	3.43	9.71		
AVERAGE		13.72	38.83		
COT ϕ	PS	16.35	46.27		
	CTL	4.54	12.85	13.69	38.75
	WES	6.91	19.56		
	NME	1.34	3.79		
AVERAGE		7.29	20.63		

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: PETROLEUM SCIENCES

		MATERIAL			
REPLICATE		ASPHALTENES			
	A	B	C	D	E
a	29.1971	25.4251	30.4130	23.0664	27.2873
b	29.5740	27.4153	28.3983	21.4001	22.6478
c	29.4544	29.3998	27.7074	21.3085	26.7020

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: CHICAGO TESTING LABS

		MATERIAL			
REPLICATE		ASPHALTENES			
	A	B	C	D	E
a	29.8802	28.3068	29.9019	22.8916	27.0005
b	31.2966	28.3192	30.2264	22.5520	27.7247
c	30.4907	28.1648	30.0932	22.0152	27.7936

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: WATERWAYS EXPERIMENT STATION

		MATERIAL			
REPLICATE		ASPHALTENES			
	A	B	C	D	E
a	30.5875	29.1315	31.2880	15.1699	26.0722
b	31.3999	28.6394	25.8907	21.2706	24.8990
c	31.8538	27.8589	29.6632	22.5257	29.9604

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

		MATERIAL			
REPLICATE		ASPHALTENES			
	A	B	C	D	E
a	31.7150	29.3138	31.1235	23.3292	29.0152
b	31.4432	29.6942	30.8510	23.0536	28.4997
c	31.1140	29.1648	30.3730	22.6972	27.9496

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: PETROLEUM SCIENCES

REPLICATE	MATERIAL SATURATES				
	A	B	C	D	E
a	16.8702	15.3385	18.5511	15.4453	16.9880
b	17.7073	15.9546	17.4571	15.7084	16.3332
c	16.6808	17.5692	18.2172	16.1380	16.8859

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL SATURATES				
	A	B	C	D	E
a	19.4921	19.2645	18.8562	17.3327	19.0666
b	19.3235	19.5192	18.7439	17.7574	18.9414
c	19.4766	19.0687	18.9152	17.2546	18.7587

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL SATURATES				
	A	B	C	D	E
a	16.2536	16.8236	17.3605	15.4922	19.2919
b	16.2281	18.0027	13.7164	14.2387	13.9370
c	17.6316	15.2583	19.6458	16.4274	17.5466

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL SATURATES				
	A	B	C	D	E
a	18.7810	18.3129	19.0236	17.4852	18.7566
b	18.5590	18.6680	18.8315	17.4230	19.1744
c	19.0228	18.6612	18.9842	18.4050	19.3048

| TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD |
LABORATORY: PETROLEUM SCIENCES

REPLICATE	MATERIAL POLARS				
	A	B	C	D	E
a	39.8546	39.3934	39.0859	36.3421	38.7789
b	38.0467	40.3288	39.4249	34.5195	41.8239
c	38.1386	38.5763	38.2513	36.7050	39.6268

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL POLARS				
	A	B	C	D	E
a	37.9934	38.2910	38.5348	34.9196	38.3494
b	36.6281	38.2714	39.2755	35.6616	38.4117
c	37.3735	37.9572	38.8729	36.4200	38.6017

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL POLARS				
	A	B	C	D	E
a	36.9013	35.4893	36.7635	37.1902	33.1912
b	36.2913	34.1936	35.3381	30.9576	35.1018
c	35.3043	36.8623	31.9691	31.9031	34.4767

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL POLARS				
	A	B	C	D	E
a	38.3494	38.4224	38.2776	40.0435	39.4896
b	39.1986	38.5615	39.7036	40.3774	39.2704
c	38.8534	37.9725	39.5627	39.7061	39.9189

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: PETROLEUM SCIENCES

REPLICATE	MATERIAL AROMATICS				
	A	B	C	D	E
a	14.0781	19.8431	17.3365	23.7266	16.9457
b	14.6721	16.3014	14.7197	28.3720	19.1952
c	15.7262	14.6190	15.8239	25.8485	16.7853

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL AROMATICS				
	A	B	C	D	E
a	12.6353	14.1369	12.7071	24.8561	14.6836
b	12.7518	13.8902	11.7542	24.0291	14.9222
c	12.6592	14.8094	12.1187	24.3192	14.8560

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL AROMATICS				
	A	B	C	D	E
a	16.2575	18.5556	14.5880	32.1431	21.4447
b	16.0801	19.1643	25.0548	33.5331	26.0621
c	15.2104	20.0205	18.7219	29.1438	18.0162

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL AROMATICS				
	A	B	C	D	E
a	11.1546	13.9518	11.5754	19.1422	12.7386
b	10.7992	13.0763	10.6139	19.1459	13.0555
c	11.0098	14.2014	11.0801	19.1917	13.8266

TABLE 2 SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
MATERIAL-ASPHALTENES

LABORATORY	REPLICATE	A	B	C	D	E
1	a	29.1971	25.4251	30.4130	23.0664	27.2873
	b	29.5740	27.4153	28.3983	21.4001	22.6478
	c	29.4544	29.3998	27.7074	21.3085	26.7020
2	a	29.8802	28.3068	29.9019	22.8916	27.9005
	b	31.2966	28.3192	30.2264	22.5520	27.7247
	c	30.4907	28.1648	30.0932	22.0152	27.7836
3	a	30.5875	29.1315	31.2880	15.1699	26.0722
	b	31.3999	28.6394	25.8907	21.2706	24.8990
	c	31.8538	27.8589	29.6632	22.5257	29.9604
4	a	31.7150	29.3138	31.1235	23.3292	29.0152
	b	31.4432	29.6942	30.8510	23.0536	28.4997
	c	31.1140	29.1648	30.3730	22.6972	27.9496

TABLE 2 SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
MATERIAL-SATURATES

LABORATORY	REPLICATE	A	B	C	D	E
1	a	16.8702	15.3385	18.5511	15.4453	16.9880
	b	17.7073	15.9546	17.4571	15.7084	16.3332
	c	16.6808	17.5692	18.2172	16.1380	16.8859
2	a	19.4921	19.2645	18.8562	17.3327	19.0666
	b	19.3235	19.5192	18.7439	17.7574	18.9414
	c	19.4766	19.0687	18.9152	17.2546	18.7587
3	a	16.2536	16.8236	17.3605	15.4922	19.2919
	b	16.2281	18.0027	13.7164	14.2387	13.9370
	c	17.6316	15.2583	19.6458	16.4274	17.5466
4	a	18.7810	18.3129	19.0236	17.4852	18.7566
	b	18.5590	18.6680	18.8315	17.4230	19.1744
	c	19.0228	18.6621	18.9842	18.4050	19.3048

TABLE 2   SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
MATERIAL-POLARS

LABORATORY	REPLICATE	A	B	C	D	E
1	a	39.8546	39.3934	39.0859	36.3421	38.7789
	b	38.0467	40.3288	39.4249	34.5195	41.8239
	c	38.1386	38.5763	38.2513	36.7050	39.6268
2	a	37.9934	38.2910	38.5348	34.9196	38.3494
	b	36.6281	38.2714	39.2755	35.6616	38.4117
	c	37.3735	37.9572	38.8729	36.4200	38.6017
3	a	36.9013	35.4893	36.7635	37.1902	33.1912
	b	36.2913	34.1936	35.3381	30.9576	35.1018
	c	35.3043	36.8623	31.9691	31.9031	34.4767
4	a	38.3494	38.4224	38.2776	40.0435	39.4896
	b	39.1986	38.5615	39.7036	40.3774	39.2704
	c	38.8534	37.9725	39.5627	39.7061	39.9189

TABLE 2   SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
MATERIAL-AROMATICS

LABORATORY	REPLICATE	A	B	C	D	E
1	a	14.0781	19.8431	17.3365	23.7266	16.9457
	b	14.6721	16.3014	14.7197	28.3720	19.1952
	c	15.7262	14.6190	15.8239	25.8485	16.7853
2	a	12.6353	14.1369	12.7071	24.8561	14.6836
	b	12.7518	13.8902	11.7542	24.0291	14.9222
	c	12.6592	14.8094	12.1187	24.3192	14.8560
3	a	16.2575	18.5556	14.5880	32.1431	21.4447
	b	16.0801	19.1643	25.0548	33.5331	26.0621
	c	15.2104	20.0205	18.7219	29.1438	18.0162
4	a	11.1546	13.9518	11.5754	19.1422	12.7386
	b	10.7992	13.0763	10.6139	19.1459	13.0555
	c	11.0098	14.2014	11.0801	19.1917	13.8266

103

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

LABORATORY	ASPHALTENES			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	29.1971	29.5740	29.4544	29.41	0.04
2	29.8802	31.2966	30.4907	30.56	0.50
3	30.5875	31.3999	31.8538	31.28	0.41
4	31.7150	31.4432	31.1140	31.42	0.09

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

LABORATORY	SATURATES			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	16.8702	17.7073	16.6808	17.09	0.30
2	19.4921	19.3235	19.4766	19.43	0.009
3	16.2536	16.2281	17.6316	16.70	0.64
4	18.7810	18.5590	19.0228	18.79	0.05

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

LABORATORY	AROMATICS			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	14.0781	14.6721	15.7262	14.83	0.70
2	12.6353	12.7518	12.6592	12.68	0.004
3	16.2575	16.0801	15.2104	15.85	0.31
4	11.1546	10.7992	11.0098	10.99	0.03

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

LABORATORY	POLARS			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	39.8546	38.0467	38.1386	38.68	1.04
2	37.9934	36.6281	37.3735	37.33	0.47
3	36.9013	36.2913	35.3043	36.17	0.65
4	38.3494	39.1986	38.8534	38.80	0.18

105

LABORATORY	ASPHALTENES			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	25.4251	27.4153	29.3998	27.42	1.41
2	28.3068	28.3192	28.1648	28.26	0.007
3	29.1315	28.6394	27.8589	28.54	0.41
4	29.3138	29.6942	29.1648	29.39	0.07

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	SATURARES			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	15.3385	15.9546	17.5692	16.29	1.33
2	19.2645	19.5192	19.0687	19.28	0.05
3	16.8236	18.0027	15.2583	16.69	1.90
4	18.3129	18.6680	18.6621	18.55	0.04

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	AROMATICS			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	19.8431	16.3014	14.6815	16.94	6.97
2	14.1369	13.8902	14.8094	14.28	0.23
3	18.5556	19.1643	20.0205	19.25	0.54
4	13.9518	13.0763	14.2014	13.74	0.35

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	POLARS			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	39.3934	40.3288	38.5763	39.43	0.77
2	38.2910	38.2714	37.9572	38.17	0.04
3	35.4893	34.1936	36.8623	35.52	1.78
4	38.4224	38.5615	37.9725	38.32	0.09

LABORATORY	ASPHALTENES			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	30.4130	28.3983	27.7074	28.84	1.97
2	29.9019	30.2264	30.0932	30.07	0.03
3	31.2880	25.8907	29.6632	28.95	7.67
4	31.1235	30.8510	30.3730	30.78	0.14

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL C

LABORATORY	SATURATES			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	17.0439	17.4571	18.2172	17.57	0.35
2	18.8562	18.7439	18.9152	18.84	0.008
3	17.3605	13.7164	19.6458	16.91	8.94
4	19.0236	18.8315	18.9842	18.95	0.01

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL C

LABORATORY	a	AROMATICS b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	15.9280	14.7197	15.8240	15.49	0.45
2	12.7071	11.7542	12.1187	12.19	0.23
3	14.5880	25.0548	18.7219	19.45	27.79
4	11.5754	10.6139	11.0801	11.09	0.23

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL C

LABORATORY	a	POLARS b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	39.0859	39.4249	38.2513	38.92	0.36
2	38.5348	39.2755	38.8729	38.89	0.14
3	36.7635	35.3381	31.9691	34.69	6.06
4	38.2776	39.7036	39.5627	39.18	0.62

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D

| LABORATORY | | ASPHALTENES | | AVERAGE x_i | WITHIN LABORATORY VARIANCE s_i^2 |
	a	b	c		
1	23.0664	21.4001	21.3085	21.87	0.81
2	22.8916	22.5520	22.0152	22.49	0.20
3	15.1699	21.2706	22.5257	19.66	15.48
4	23.3292	23.0536	22.6972	23.03	0.10

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D

| LABORATORY | | SATURATES | | AVERAGE x_i | WITHIN LABORATORY VARIANCE s_i^2 |
	a	b	c		
1	15.4453	15.7084	16.1380	15.73	0.16
2	17.3327	17.7574	17.2546	17.45	0.07
3	15.4922	14.2387	16.4274	15.39	1.21
4	17.4852	17.4230	18.4050	17.77	0.30

TABLE 3 BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D				AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
LABORATORY	a	AROMATICS b	c		
1	26.5940	28.3720	25.8485	26.94	1.68
2	24.8561	24.0291	24.3192	24.40	0.18
3	32.1431	33.5331	29.1438	31.61	5.03
4	19.1422	19.1459	19.1917	19.16	0.0008

TABLE 3 BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D				AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
LABORATORY	a	POLARS b	c		
1	36.3421	34.5195	36.7050	35.86	1.37
2	34.9196	35.6616	36.4200	35.67	0.56
3	37.1902	30.9576	31.9031	33.35	11.28
4	40.0435	40.3774	39.7061	40.04	0.11

111

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	ASPHALTENES			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	27.2873	22.6478	26.7020	25.55	6.38
2	27.9005	27.7247	27.7836	27.80	0.008
3	26.0722	24.8990	29.9604	26.98	7.02
4	29.0152	28.4997	27.9496	28.49	0.28

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	SATURATES			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	16.9880	16.3332	16.8859	16.74	0.12
2	19.0666	18.9414	18.7587	18.92	0.02
3	19.2919	13.9370	17.5466	16.93	7.46
4	18.7566	19.1744	19.3048	19.08	0.08

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	AROMATICS			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	16.9457	19.1952	16.7853	17.64	1.82
2	14.6836	14.9222	14.8560	14.82	0.02
3	21.4447	26.0621	18.0162	21.84	16.30
4	12.7386	13.0555	13.8266	13.21	0.31

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	POLARS			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	38.7789	41.8239	39.6268	40.08	2.47
2	38.3494	38.4117	38.6017	38.45	0.02
3	33.1912	35.1018	34.4767	34.26	0.95
4	39.4896	39.2704	39.9189	39.56	0.11

TABLE 4 AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

MATERIAL	AVERAGE ASPHALTENES	COMPONENTS OF VARIANCE		VARIANCE	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
D	21.71	4.01	0.85	4.01	4.86
E	27.21	3.42	0.46	3.42	3.88
B	28.41	1.17	0.24	1.17	1.41
C	29.46	1.99	0.82	1.99	2.81
A	30.67	0.26	0.76	0.26	1.02

TABLE 4 AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

MATERIAL	AVERAGE SATURATES	COMPONENTS OF VARIANCE		VARIANCE	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
D	16.55	0.50	1.37	0.50	1.87
B	17.67	0.73	1.95	0.73	2.68
E	17.92	1.92	0.93	1.92	2.85
A	18.00	0.25	1.65	0.25	1.90
C	18.07	2.33	0.21	2.33	2.54

TABLE 4 AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

MATERIAL	AVERAGE AROMATICS	COMPONENTS OF VARIANCE		VARIANCE	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	13.59	0.26	4.66	0.26	4.92
C	14.56	7.18	11.75	7.18	18.93
B	16.05	2.02	5.83	2.02	7.85
E	16.88	4.61	12.76	4.62	17.37
D	25.53	1.72	26.36	1.72	28.08

TABLE 4 AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

MATERIAL	AVERAGE POLARS	COMPONENTS OF VARIANCE		VARIANCE	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
D	36.23	3.33	6.64	3.33	9.97
A	37.75	0.59	1.35	0.59	1.94
B	37.86	0.67	2.53	0.67	3.20
C	37.92	1.80	4.05	1.80	5.85
E	38.09	0.89	6.68	0.89	7.57

TABLE 5   AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIATION
FOR ALL MATERIALS

MATERIAL	AVERAGE ASPHALT.	STANDARD DEVIATIONS		COEFFICIENTS OF VARIATION	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
D	21.76	2.04	2.22	9.38	10.20
E	27.21	1.85	1.97	6.80	7.24
B	28.40	0.69	0.98	2.43	3.45
C	29.66	1.57	1.58	5.29	5.33
A	30.67	0.51	1.00	1.66	3.26

TABLE 5   AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIATION
FOR ALL MATERIALS

MATERIAL	AVERAGE SATURATE	STANDARD DEVIATIONS		COEFFICIENTS OF VARIATION	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
D	16.59	0.66	1.22	3.98	7.35
B	17.70	0.91	1.62	5.14	9.15
E	17.92	1.39	1.69	7.76	9.43
A	18.00	0.50	1.37	2.78	7.61
C	18.20	1.52	1.56	8.35	8.57

TABLE 5 AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIATION
FOR ALL MATERIALS

| MATERIAL | AVERAGE AROMATIC | STANDARD DEVIATIONS | | COEFFICIENTS OF VARIATION | |
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	13.59	0.51	2.22	3.75	16.34
C	14.67	2.74	4.41	18.68	30.06
B	16.05	1.44	2.80	8.97	17.45
E	16.88	2.15	4.17	12.74	24.70
D	25.28	1.66	5.30	6.57	20.97

TABLE 5 AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIATION
FOR ALL MATERIALS

| MATERIAL | AVERAGE POLARS | STANDARD DEVIATIONS | | COEFFICIENTS OF VARIATION | |
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
D	36.33	1.89	3.18	5.20	8.75
C	37.47	2.00	2.63	5.34	7.02
A	37.75	0.77	1.39	2.04	3.68
B	37.85	0.84	1.78	2.22	4.70
E	38.09	0.94	2.75	2.47	7.22

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: PETEOLEUM SCIENCES

REPLICATE	MATERIAL X min				
	A	B	C	D	E
a	1.18	1.46	1.80	2.23	1.13
b	1.24	1.56	1.18	2.13	1.76
c	1.54	1.35	1.15	2.21	1.58

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL X min				
	A	B	C	D	E
a	1.46	1.54	1.74	2.22	1.71
b	1.46	1.35	1.75	2.02	1.80
c	1.58	1.53	1.49	2.15	1.83

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL X min				
	A	B	C	D	E
a	1.99	2.36	1.76	2.64	1.83
b	1.61	1.82	1.96	1.92	2.25
c	2.05	1.92	1.80	2.35	2.16

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL X min				
	A	B	C	D	E
a	1.91	1.91	1.93	2.17	1.78
b	1.89	1.92	1.87	2.29	1.96
c	1.99	1.89	1.68	2.29	1.99

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: PETROLEUM SCIENCES

REPLICATE	MATERIAL Pa				
	A	B	C	D	E
a	0.45	0.50	0.60	0.68	0.46
b	0.49	0.56	0.53	0.69	0.61
c	0.53	0.53	0.49	0.66	0.61

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL Pa				
	A	B	C	D	E
a	0.62	0.62	0.59	0.68	0.62
b	0.62	0.63	0.60	0.68	0.62
c	0.61	0.63	0.61	0.67	0.61

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL Pa				
	A	B	C	D	E
a	0.63	0.63	0.64	0.71	0.69
b	0.63	0.67	0.64	0.72	0.63
c	0.61	0.66	0.64	0.69	0.64

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL Pa				
	A	B	C	D	E
a	0.59	0.61	0.60	0.68	0.61
b	0.60	0.60	0.60	0.68	0.61
c	0.59	0.60	0.61	0.68	0.61

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: PETROLEUM SCIENCES

REPLICATE	MATERIAL P				
	A	B	C	D	E
a	2.18	2.46	2.80	3.23	2.13
b	2.24	2.56	2.18	3.13	2.76
c	2.54	2.35	2.15	3.21	2.58

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL P				
	A	B	C	D	E
a	2.46	2.54	2.74	3.22	2.71
b	2.46	2.35	2.75	3.02	2.80
c	2.58	2.53	2.49	3.15	2.83

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL P				
	A	B	C	D	E
a	2.99	3.36	2.76	3.64	2.83
b	2.61	2.82	2.96	2.92	3.25
c	3.05	2.92	2.80	3.35	3.16

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL P				
	A	B	C	D	E
a	2.91	2.91	2.93	3.17	2.78
b	2.89	2.92	2.87	3.29	2.96
c	2.99	2.89	2.68	3.29	2.99

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: PETROLEUM SCIENCES

REPLICATE	MATERIAL Po				
	A	B	C	D	E
a	1.19	1.24	1.11	1.05	1.15
b	1.15	1.14	1.03	0.97	1.09
c	1.20	1.10	1.10	1.08	0.99

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL Po				
	A	B	C	D	E
a	0.95	0.96	1.11	1.04	1.04
b	0.94	0.86	1.11	0.96	1.08
c	1.01	0.93	0.97	1.03	1.11

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL Po				
	A	B	C	D	E
a	1.11	1.24	1.00	1.04	0.89
b	0.97	0.93	1.06	0.83	1.20
c	1.20	0.98	1.01	1.04	1.15

TABLE 1   DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL Po				
	A	B	C	D	E
a	1.19	1.14	1.17	1.00	1.07
b	1.17	1.16	1.14	1.06	1.15
c	1.23	1.14	1.04	1.04	1.16

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: PETROLEUM SCIENCES

REPLICATE	MATERIAL COT				
	A	B	C	D	E
a	0.73	1.51	1.49	2.16	0.87
b	1.02	1.45	1.06	2.21	1.60
c	1.27	1.53	1.13	2.32	1.49

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL COT				
	A	B	C	D	E
a	1.54	1.55	1.39	2.01	1.57
b	1.54	1.63	1.41	2.11	1.61
c	1.51	1.58	1.49	2.14	1.55

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL COT				
	A	B	C	D	E
a	1.63	1.62	1.72	2.28	2.03
b	1.59	1.93	1.76	2.46	1.63
c	1.48	1.90	1.70	2.24	1.61

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL COT				
	A	B	C	D	E
a	1.42	1.55	1.52	2.15	1.53
b	1.45	1.50	1.50	2.07	1.53
c	1.42	1.55	1.51	2.16	1.51

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: PETROLEUM SCIENCES

REPLICATE	MATERIAL To				
	A	B	C	D	E
a	1.40	0.28	1.85	2.06	1.09
b	1.10	1.10	1.26	2.15	1.61
c	1.20	0.45	0.73	1.45	1.79

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: CHICAGO TESTING LABS

REPLICATE	MATERIAL To				
	A	B	C	D	E
a	1.59	1.77	1.89	2.36	1.78
b	1.61	1.54	1.89	2.13	1.78
c	1.66	1.81	1.63	1.94	1.84

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: WATERWAYS EXPERIMENT STATION

REPLICATE	MATERIAL To				
	A	B	C	D	E
a	2.11	2.57	1.85	3.07	2.18
b	1.82	2.02	2.02	2.07	2.41
c	2.19	2.07	1.93	2.31	2.43

TABLE 1 DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
LABORATORY: NEW MEXICO ENGINEERING RESEARCH INSTITUTE

REPLICATE	MATERIAL To				
	A	B	C	D	E
a	1.96	1.91	1.91	2.21	1.90
b	1.94	1.97	1.92	2.36	2.03
c	2.02	1.85	1.82	2.27	2.13

TABLE 2 SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
MATERIAL-X min

LABORATORY	REPLICATE	A	B	C	D	E
1	a	1.18	1.46	1.80	2.23	1.13
	b	1.24	1.56	1.18	2.13	1.76
	c	1.54	1.35	1.15	2.21	1.58
2	a	1.46	1.54	1.74	2.22	1.71
	b	1.46	1.35	1.75	2.02	1.80
	c	1.58	1.53	1.49	2.15	1.83
3	a	1.99	2.36	1.76	2.64	1.83
	b	1.61	1.82	1.96	1.92	2.25
	c	2.05	1.92	1.80	2.35	2.16
4	a	1.91	1.91	1.93	2.17	1.78
	b	1.89	1.92	1.87	2.29	1.96
	c	1.99	1.89	1.68	2.29	1.99

TABLE 2 SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
MATERIAL-Po.

LABORATORY	REPLICATE	A	B	C	D	E
1	a	1.19	1.24	1.11	1.05	1.15
	b	1.15	1.14	1.03	0.97	1.09
	c	1.20	1.10	1.10	1.08	0.99
2	a	0.95	0.96	1.11	1.04	1.04
	b	0.94	0.86	1.11	0.96	1.08
	c	1.01	0.93	0.97	1.03	1.11
3	a	1.11	1.24	1.00	1.04	0.89
	b	0.97	0.93	1.06	0.83	1.20
	c	1.20	0.98	1.01	1.04	1.15
4	a	1.19	1.14	1.17	1.00	1.07
	b	1.17	1.16	1.14	1.06	1.15
	c	1.23	1.14	1.04	1.04	1.16

TABLE 2 SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

MATERIAL-P

LABORATORY	REPLICATE	A	B	C	D	E
1	a	2.18	2.46	2.80	3.23	2.13
	b	2.24	2.56	2.18	3.13	2.76
	c	2.54	2.35	2.15	3.21	2.58
2	a	2.46	2.54	2.74	3.22	2.71
	b	2.46	2.35	2.75	3.02	2.80
	c	2.58	2.53	2.49	3.15	2.83
3	a	2.99	3.36	2.76	3.64	2.83
	b	2.61	2.82	2.96	2.92	3.25
	c	3.05	2.92	2.80	3.35	3.16
4	a	2.91	2.91	2.93	3.17	2.78
	b	2.89	2.92	2.87	3.29	2.96
	c	2.99	2.89	2.68	3.29	2.99

TABLE 2 SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD

MATERIAL-Pa

LABORATORY	REPLICATE	A	B	C	D	E
1	a	0.45	0.50	0.60	0.68	0.46
	b	0.49	0.56	0.53	0.69	0.61
	c	0.53	0.53	0.49	0.66	0.61
2	a	0.62	0.62	0.59	0.68	0.62
	b	0.62	0.63	0.60	0.68	0.62
	c	0.61	0.63	0.61	0.67	0.61
3	a	0.63	0.63	0.64	0.71	0.69
	b	0.63	0.67	0.64	0.72	0.63
	c	0.61	0.66	0.64	0.69	0.64
4	a	0.59	0.61	0.60	0.68	0.61
	b	0.60	0.60	0.60	0.68	0.61
	c	0.59	0.60	0.61	0.68	0.61

TABLE 2 SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
MATERIAL-COT

LABORATORY	REPLICATE	A	B	C	D	E
1	a	0.73	1.51	1.49	2.16	0.87
	b	1.02	1.45	1.06	2.21	1.60
	c	1.27	1.53	1.13	2.32	1.49
2	a	1.54	1.55	1.39	2.01	1.57
	b	1.54	1.63	1.41	2.11	1.61
	c	1.51	1.58	1.49	2.14	1.55
3	a	1.63	1.62	1.72	2.28	2.03
	b	1.59	1.93	1.76	2.46	1.63
	c	1.48	1.90	1.70	2.24	1.61
4	a	1.42	1.55	1.52	2.15	1.53
	b	1.45	1.50	1.50	2.07	1.53
	c	1.42	1.55	1.51	2.16	1.51

TABLE 2 SUMMARY DATA SHEET FOR INTERLABORATORY TEST PROGRAM FOR ASTM METHOD
MATERIAL-To

LABORATORY	REPLICATE	A	B	C	D	E
1	a	1.40	0.28	1.85	2.06	1.09
	b	1.10	1.10	1.26	2.15	1.61
	c	1.20	0.45	0.73	1.45	1.79
2	a	1.59	1.77	1.89	2.36	1.78
	b	1.61	1.54	1.89	2.13	1.78
	c	1.66	1.81	1.63	1.94	1.84
3	a	2.11	2.57	1.85	3.07	2.18
	b	1.82	2.02	2.02	2.07	2.41
	c	2.19	2.07	1.93	2.31	2.43
4	a	1.96	1.91	1.91	2.21	1.90
	b	1.94	1.97	1.92	2.36	2.03
	c	2.02	1.85	1.82	2.27	2.13

TABLE 3 BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

LABORATORY	X min			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.18	1.24	1.54	1.32	0.0372
2	1.46	1.46	1.58	1.50	0.0048
3	1.99	1.61	2.05	1.88	0.0569
4	1.91	1.89	1.99	1.93	0.0028

TABLE 3 BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

LABORATORY	Pa			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	0.45	0.49	0.53	0.490	0.0016
2	0.62	0.62	0.61	0.617	0.00003
3	0.63	0.63	0.61	0.623	0.0001
4	0.59	0.60	0.59	0.593	0.00003

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

| LABORATORY | p | | | AVERAGE x_i | WITHIN LABORATORY VARIANCE s_i^2 |
	a	b	c		
1	2.18	2.24	2.54	2.32	0.0372
2	2.46	2.46	2.58	2.50	0.0048
3	2.99	2.61	3.05	2.88	0.0569
4	2.91	2.89	2.99	2.93	0.0028

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

| LABORATORY | Po | | | AVERAGE x_i | WITHIN LABORATORY VARIANCE s_i^2 |
	a	b	c		
1	1.19	1.15	1.20	1.18	0.0007
2	0.95	0.94	1.01	0.97	0.0014
3	1.11	0.97	1.20	1.09	0.0134
4	1.19	1.17	1.23	1.20	0.0009

LABORATORY	a	COT b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	0.73	1.02	1.27	1.01	0.0730
2	1.54	1.54	1.51	1.53	0.0003
3	1.63	1.59	1.48	1.57	0.0060
4	1.42	1.45	1.42	1.43	0.0003

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL A

LABORATORY	a	T_b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	1.40	1.10	1.20	1.23	0.0233
2	1.59	1.61	1.66	1.62	0.0013
3	2.11	1.82	2.19	2.04	0.0379
4	1.96	1.94	2.02	1.97	0.0017

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	a	X min b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	1.46	1.56	1.35	1.46	0.0110
2	1.54	1.35	1.53	1.47	0.0114
3	2.36	1.82	1.92	2.03	0.0825
4	1.91	1.92	1.89	1.91	0.0002

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	a	Pa b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	0.50	0.56	0.53	0.530	0.0009
2	0.62	0.63	0.63	0.627	0.00003
3	0.63	0.67	0.66	0.653	0.0004
4	0.61	0.60	0.60	0.603	0.00003

130

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	P			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	2.46	2.56	2.35	2.46	0.0110
2	2.54	2.35	2.53	2.47	0.0114
3	3.36	2.82	2.92	3.03	0.0825
4	2.91	2.92	2.89	2.91	0.0002

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	Po			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.24	1.14	1.10	1.16	0.0052
2	0.96	0.86	0.93	0.92	0.0026
3	1.24	0.93	0.98	1.05	0.0277
4	1.14	1.16	1.14	1.15	0.0001

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	COT			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.51	1.45	1.53	1.50	0.0017
2	1.55	1.63	1.58	1.59	0.0016
3	1.62	1.93	1.90	1.82	0.0292
4	1.55	1.50	1.55	1.53	0.0008

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL B

LABORATORY	T_b			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	0.28	1.10	0.45	0.61	0.1873
2	1.77	1.54	1.81	1.71	0.0212
3	2.57	2.02	2.07	2.22	0.0925
4	1.91	1.97	1.85	1.91	0.0036

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL C

LABORATORY		X min		AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.80	1.18	1.15	1.38	0.1346
2	1.74	1.75	1.49	1.66	0.0217
3	1.76	1.96	1.80	1.84	0.0112
4	1.93	1.87	1.68	1.83	0.0170

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL C

LABORATORY		Pa		AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	0.60	0.53	0.49	0.540	0.0031
2	0.59	0.60	0.61	0.600	0.0001
3	0.64	0.64	0.64	0.640	0.0000
4	0.60	0.60	0.61	0.603	0.00003

133

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL C

LABORATORY	P			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	2.80	2.18	2.15	2.38	0.1346
2	2.74	2.75	2.49	2.66	0.0217
3	2.76	2.96	2.80	2.84	0.0112
4	2.93	2.87	2.68	2.83	0.0170

LABORATORY	Po			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.11	1.03	1.10	1.08	0.0019
2	1.11	1.11	0.97	1.06	0.0065
3	1.00	1.06	1.01	1.02	0.0010
4	1.17	1.14	1.04	1.12	0.0046

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL C

LABORATORY	COT			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.49	1.06	1.13	1.23	0.0532
2	1.39	1.41	1.49	1.43	0.0028
3	1.72	1.76	1.70	1.73	0.0009
4	1.52	1.50	1.51	1.51	0.0001

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL C

LABORATORY	TP			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.85	1.26	0.73	1.28	0.3139
2	1.89	1.89	1.63	1.80	0.0225
3	1.85	2.02	1.93	1.93	0.0072
4	1.91	1.92	1.82	1.88	0.0030

TABLE 3 BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D

LABORATORY	a	X min b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	2.23	2.13	2.21	2.19	0.0028
2	2.22	2.02	2.15	2.13	0.0103
3	2.64	1.92	2.35	2.30	0.1312
4	2.17	2.29	2.29	2.25	0.0048

TABLE 3 BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D

LABORATORY	a	Pa b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	0.68	0.69	0.66	0.677	0.0002
2	0.68	0.68	0.67	0.677	0.00003
3	0.71	0.72	0.69	0.707	0.0002
4	0.68	0.68	0.68	0.680	0.0000

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D

LABORATORY	P			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	3.23	3.13	3.21	3.19	0.0028
2	3.22	3.02	3.15	3.13	0.0103
3	3.64	2.92	3.35	3.30	0.1312
4	3.17	3.29	3.29	3.25	0.0048

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D

LABORATORY	Po			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.05	0.97	1.08	1.03	0.0032
2	1.04	0.96	1.03	1.01	0.0019
3	1.04	0.83	1.04	0.97	0.0147
4	1.00	1.06	1.04	1.03	0.0009

TABLE 3 BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D					
LABORATORY		COT		AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	2.16	2.21	2.32	2.23	0.0067
2	2.01	2.11	2.14	2.09	0.0046
3	2.28	2.46	2.24	2.33	0.0137
4	2.15	2.07	2.16	2.13	0.0024

TABLE 3 BETWEEN AND WITHIN ANALYSIS FOR MATERIAL D					
LABORATORY		TP b		AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a		c		
1	2.06	2.15	1.45	1.89	0.1450
2	2.36	2.13	1.94	2.14	0.0442
3	3.07	2.07	2.31	2.48	0.2725
4	2.21	2.36	2.27	2.28	0.0057

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	a	X min b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	1.13	1.76	1.58	1.49	0.1053
2	1.71	1.80	1.83	1.78	0.0039
3	1.83	2.25	2.16	2.08	0.0489
4	1.78	1.96	1.99	1.91	0.0129

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	a	Pa b	c	AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
1	0.46	0.61	0.61	0.560	0.0075
2	0.62	0.62	0.61	0.617	0.00003
3	0.69	0.63	0.64	0.653	0.0010
4	0.61	0.61	0.61	0.610	0.0000

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	P			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	2.13	2.76	2.58	2.49	0.1053
2	2.71	2.80	2.83	2.78	0.0039
3	2.83	3.25	3.16	3.08	0.0489
4	2.78	2.96	2.99	2.91	0.0129

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	Po			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.15	1.09	0.99	1.08	0.0065
2	1.04	1.08	1.11	1.08	0.0012
3	0.89	1.20	1.15	1.08	0.0277
4	1.07	1.15	1.16	1.13	0.0024

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	COT			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	0.9	1.60	1.49	1.32	0.1549
2	1.57	1.61	1.55	1.58	0.0009
3	2.03	1.63	1.61	1.76	0.0561
4	1.53	1.53	1.51	1.52	0.0001

TABLE 3   BETWEEN AND WITHIN ANALYSIS FOR MATERIAL E

LABORATORY	T_D			AVERAGE x_i	WITHIN LABORATORY VARIANCE s_i^2
	a	b	c		
1	1.09	1.61	1.79	1.50	0.1321
2	1.78	1.78	1.84	1.80	0.0012
3	2.18	2.41	2.43	2.34	0.0193
4	1.90	2.03	2.13	2.02	0.0133

TABLE 4 AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

		COMPONENTS OF VARIANCE		VARIANCE	
MATERIAL	AVERAGE X min	WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	1.66	0.0254	0.0790	0.0254	0.1044
C	1.68	0.0461	0.0308	0.0461	0.0769
B	1.72	0.0263	0.0786	0.0263	0.1049
E	1.82	0.0428	0.0477	0.0428	0.0905
D	2.22	0.0373	-0.0070	0.0373	0.0303

TABLE 4 AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

		COMPONENTS OF VARIANCE		VARIANCE	
MATERIAL	AVERAGE Pa	WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	0.581	0.0004	0.0037	0.0004	0.0041
C	0.596	0.0008	0.0014	0.0008	0.0022
B	0.603	0.0003	0.0027	0.0003	0.0030
E	0.610	0.0021	0.0008	0.0021	0.0029
D	0.686	0.0001	0.0002	0.0001	0.0003

TABLE 4   AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

| MATERIAL | AVERAGE P | COMPONENTS OF VARIANCE | | VARIANCE | |
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	2.66	0.0254	0.0790	0.0254	0.1044
C	2.68	0.0461	0.0308	0.0461	0.0769
B	2.72	0.0263	0.0786	0.0263	0.1049
E	2.82	0.0428	0.0477	0.0428	0.0905
D	3.22	0.0373	-0.0070	0.0373	0.0303

TABLE 4   AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

| MATERIAL | AVERAGE Po | COMPONENTS OF VARIANCE | | VARIANCE | |
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
D	1.01	0.0052	-0.0009	0.0052	0.0043
B	1.07	0.0089	0.0095	0.0089	0.0184
C	1.07	0.0035	0.0005	0.0035	0.0040
E	1.09	0.0095	-0.0026	0.0095	0.0069
A	1.11	0.0041	0.0096	0.0041	0.0137

143

TABLE 4 AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

| MATERIAL | AVERAGE COT | COMPONENTS OF VARIANCE | | VARIANCE | |
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	1.39	0.0199	0.0594	0.0199	0.0793
C	1.48	0.0143	0.0380	0.0143	0.0523
E	1.55	0.0530	0.0152	0.0530	0.0682
B	1.61	0.0083	0.0182	0.0083	0.0265
D	2.20	0.0069	0.0093	0.0069	0.0162

TABLE 4 AVERAGES, COMPONENTS OF VARIANCE, AND VARIANCES FOR ALL MATERIALS

| MATERIAL | AVERAGE To | COMPONENTS OF VARIANCE | | VARIANCE | |
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
B	1.61	0.0762	0.4653	0.0762	0.5415
A	1.72	0.0161	0.1329	0.0161	0.1490
C	1.72	0.0867	0.0610	0.0867	0.1477
E	1.92	0.0415	0.1119	0.0415	0.1534
D	2.20	0.1169	0.0225	0.1169	0.1394

TABLE 5 AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIANCE
FOR ALL MATERIALS

MATERIAL	AVERAGE X min	STANDARD DEVIATIONS		COEFFICIENTS OF VARIANCE	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	1.66	0.1594	0.3231	0.0960	0.1946
C	1.68	0.2147	0.2773	0.1278	0.1651
B	1.72	0.1622	0.3239	0.0943	0.1883
E	1.82	0.2069	0.3008	0.1137	0.1653
D	2.22	0.1931	0.1741	0.0870	0.0784

TABLE 5 AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIANCE
FOR ALL MATERIALS

MATERIAL	AVERAGE Pa	STANDARD DEVIATIONS		COEFFICIENTS OF VARIANCE	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	0.581	0.0200	0.0640	0.0344	0.1102
C	0.596	0.0283	0.0469	0.0475	0.0787
B	0.603	0.0173	0.0548	0.0287	0.0909
E	0.610	0.0458	0.0539	0.0751	0.0884
D	0.686	0.0100	0.0173	0.0146	0.0252

TABLE 5   AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIATION
FOR ALL MATERIALS

MATERIAL	AVERAGE P	STANDARD DEVIATIONS		COEFFICIENTS OF VARIATION	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	2.66	0.1594	0.3231	0.0599	0.1215
C	2.68	0.2147	0.2773	0.0801	0.1035
B	2.72	0.1622	0.3239	0.0596	0.1191
E	2.82	0.2069	0.3008	0.0734	0.1067
D	3.22	0.1931	0.1741	0.0600	0.0541

TABLE 5   AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIATION
FOR ALL MATERIALS

MATERIAL	AVERAGE Po	STANDARD DEVIATIONS		COEFFICIENTS OF VARIATION	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
D	1.01	0.0721	0.0656	0.0714	0.0650
B	1.07	0.0943	0.1356	0.0881	0.1267
C	1.07	0.0592	0.0632	0.0553	0.0591
E	1.09	0.0975	0.0831	0.0894	0.0762
A	1.11	0.0640	0.1170	0.0577	0.1054

TABLE 5   AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIATION
FOR ALL MATERIALS

MATERIAL	AVERAGE COT	STANDARD DEVIATIONS		COEFFICIENTS OF VARIATION	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
A	1.39	0.1411	0.2816	0.1015	0.2026
C	1.48	0.1196	0.2287	0.0808	0.1545
E	1.55	0.2302	0.2612	0.1485	0.1685
B	1.61	0.0911	0.1628	0.0566	0.1011
D	2.20	0.0831	0.1273	0.0378	0.0579

TABLE 5   AVERAGES, STANDARD DEVIATIONS, AND COEFFICIENTS OF VARIATION
FOR ALL MATERIALS

MATERIAL	AVERAGE To	STANDARD DEVIATIONS		COEFFICIENTS OF VARIATION	
		WITHIN LABORATORY	BETWEEN LABORATORY	WITHIN LABORATORY	BETWEEN LABORATORY
B	1.61	0.2760	0.7359	0.1714	0.4571
A	1.72	0.1269	0.3860	0.0738	0.2244
C	1.72	0.2944	0.3843	0.1712	0.2234
E	1.92	0.2037	0.3917	0.1061	0.2040
D	2.20	0.3419	0.3734	0.1554	0.1697

147

(The reverse of this page is blank.)

APPENDIX B
CLAY-GEL TEST PROCEDURE

AIR FORCE ENGINEERING SERVICES CENTER

TENTATIVE TEST PROCEDURE (AFESC-1)

**CHARACTERISTIC GROUPS IN ASPHALT BINDER BY THE
CLAY-GEL ADSORPTION CHROMATOGRAPHIC METHOD**

Draft of Tentative Test Method (AFESC-1) for

CHARACTERISTIC GROUPS IN ASPHALT BINDER BY THE
CLAY-GEL ADSORPTION CHROMATOGRAPHIC METHOD

B1.0   SCOPE.

B1.1   This method covers a procedure for separating bituminous materials into four generic fractions; asphaltenes, saturates, aromatics, and polars and the determination of the amounts of each. When the sample contains more than one percent insolubles in n-pentane (asphaltenes), the method provides for removal of the insoluble material prior to chromatographic fractionation, and the determination of the amount present.

> Note 1 - (Clay choice important). When clays other than the original Florex S clay are used for the determination of polar compounds, the results may not necessarily be equivalent.

B2.0   APPLICABLE DOCUMENTS.

B2.1   ASTM Standards :

D 216, "Standard Method for Distillation of Natural Gaso·ine".[1]

D 329, "Standard Specification for Acetone".[2]

D 1159, "Standard Test Method for Bromine Number of Petroleum Distillates and Commercial Aliphatic Olefins by Electrometric Titration".[3]

D 2226, "Recommended Practice for Description of Types of Petroleum Extender Oils".[4]

---

1. 1982 Annual Book of Standards, Volume 05.01.

2. 1981 Annual Book of Standards, Volume 06.03.

3. 1984 Annual Book of Standards, Volume 05.01.

4. 1982 Annual Book of Standandards, Volumes 05.02 and 09.01.

B3.0 SUMMARY OF METHOD.

B3.1 The sample is dissolved in n-pentane and filtered to remove asphaltenes (Note 2). The filtered solution is rotary evaporated to remove the n-pentane solvent. Solvent is then charged onto two glass percolation columns connected in series, containing clay in the upper sections and silica gel (plus clay) in the lower section (see Figure B1). The effluents from this and succeeding chargings are separately collected in specified volumes.

Note 2- The insoluble matter (asphaltenes) must be removed from the sample prior to charging the column if the asphalt samples contain more than one weight percent n-pentane insolubles, as determined in Section B9.

The upper (clay) section is removed from the lower section and washed further with n-pentane. An acetone-toluene mixture 63 percent acetone to 37 percent toluene by volume, is then charged to the clay section. Methylene chloride is used as a final eluent to strip polar compounds from the clay column.

The lower (silica) column is charged with a toluene-acetone mixture of 80 percent toluene to 20 percent acetone by volume. Pure toluene is used as a final eluent to strip aromatics from the silica column

B3.2 The solvents are completely removed from the recovered effluents by the use of a rotary evaporator and the residues are weighed and calculated as saturates, aromatics, and polar compounds. See Figure B2 for a flow chart of the procedure.

B4.0 SIGNIFICANCE AND USE.

B4.1 Concentrations of characteristic hydrocarbon groups as determined by this method are used to classify bituminous materials. Compatibility and certain finished product properties can often be correlated with the composition as determined by this method.

B5.0   DESCRIPTION OF TERMS.

B5.1   The following descriptions refer to the hydrocarbon types and structural groups as measured by this method :

B5.1.1   Asphaltenes, or n-pentane insolubles. Insoluble matter that can be separated from the asphalt.

B5.1.2   Polar compounds. Material retained on adsorbent clay after percolation of the sample in a n-pentane eluent under the conditions specified.

> Note 3- For classification purposes, the group "polar aromatics" or "resins" is the same as "polar compounds".

B5.1.3   Aromatics. Material that on percolation, passes through a column of adsorbent clay in a n-pentane eluent, but adsorbs on silica gel under the conditions specified.

B5.1.4   Saturates. Material, that, on percolation in a n-pentane eluent, is not adsorbed on either clay or silica gel under the conditions specified.

B6.0 APPARATUS.

B6.1   Two clay-gel columns, 40 mm diameter by 318 mm from top of column to top of fritted glass. 70 mm from top of fritted glass to bottom of column, constructed as illustrated in Figure B1.

B6.2   Conical flask, wide mouthed, graduated 500 and 1000 ml capacity. Six required, see Section B10 for identification details.

B6.3   Convection oven.

B6.4   Filter funnel, Buchner-type, glass 150 ml capacity, 60 mm diameter, fine porosity filter disk.

B6.5   Filter funnel, long stem 100 mm diameter (optional).

B6.6   Filter flask, 500 ml.

B6.7   Hot-water bath with nitrogen jets.

B6.8   Hot plate.

B6.9   Rotary evaporator, including boiling flask, collecting flask, water condenser, nitrogen jet, hot water bath and vacuum.

B6.10   Vacuum and vacuum tank.

B7.0   REAGENTS AND MATERIALS.

B7.1   Acetone, reagent grade.

B7.2   Acetone-toluene mixtures (20 acetone to 80 toluene and 63 acetone to 37 toluene by volume).

B7.3   Clay adsorbent, 30 to 60 mesh Attapulgus. Clay quality must be determined using the azobenzene activity test according to ASTM 2007[5]. The azobenzene activity test measures the adsorptive characteristics of the clay. Azobenzene activity value should be 26 to 29. Clay outside of these limits should be discarded.

B7.4   Methylene chloride, reagent grade.

B7.5   Molecular sieves 8-12 mesh. Approximately 20g are placed in reagent containers to remove water. Sieves should be prevented from entering columns during test.

B7.6   Pentane, reagent grade.

B7.7   Silica gel, activated, 28 to 200 mesh. This material is available from Forcoven Products, Inc., Box 1488, Humble, TX, 77338 and specifically prepared for ASTM D 2007[5].

---

5. D 2007-80, "Standard Test Method for Characteristic Groups in Rubber Extender and Processing Oils by the Clay-Gel Adsorption Chromatographic Method". 1985 Annual Book of ASTM Standards, Volume 09.01

B7.8   Toluene, reagent grade.

B7.9   Trichloroethylene, technical grade.

B8.0   PREPARATION OF SAMPLE.

B8.1   If the n-pentane insolubles content is known to be above one percent by weight, or if there is some uncertainty that this content is not below one percent by weight, remove and determine any such insolubles as described in Section B9.

B8.2   If it is certain that the n-pentane insolubles content is less than one weight percent, proceed to Section B10.

B9.0   INSOLUBLES REMOVAL.

B9.1   Weigh 2.5 ± 0.05 g of the sample to the nearest 0.0001g in a preweighed 250 ml conical flask; add 62.5 ml n-pentane (25 ml/g of sample) and mix well. Allow the sample to stand overnight. Use a glass stirring rod to gently break up the clump of asphalt. Start at the edge of the clump and work inward to solubilize as much asphalt as possible.

B9.2   Set up a filtering assembly using a 500 ml suction flask and a 150 ml capacity glass Buchner funnel. Filter the sample-n-pentane mixture. Rinse out the conical flask twice with 10-20 ml n-pentane and pour the two rinse solvents through the filtering funnel. This flask should be retained for later weighing; see Section B11.3

B9.3   Add approximately 500 ml of pentane solvent, in four or five equal portions, to the filter funnel just when the precipitate collected in the funnel appears dry on the surface. Wash the inner walls of the funnel with 10-20 ml of pentane solvent. Place aluminum foil (or other suitable material) over the top of funnel and remove funnel from filtering flask (Note 4). Pour filtrate into 1000 ml boiling flask and place in rotary evaporator to remove solvent, as described in Section 9.4. Return Buchner to filtering flask and wash twice more, as before, with 500 ml of pentane. Rotary evaporate after each wash.

The covered Buchner should be placed in a drying oven for one hour in
order to remove any water that remains on the funnel or in the
asphaltenes, removed and placed in a dessicator to cool, and then weighed.
This procedure should be repeated until a constant weight is obtained.
This precipitated material remaining on the filter is the asphaltene
fraction.

    Note 4- The Buchner is covered to decrease the possibility of losing
    any of the drying asphaltenes into the fume hood.

B9.4   Adjust the boiling flasks containing the filtrate over the warm
water bath of the rotary evaporator such that the flasks just touch the
water. The flask can be lowered as solvent evaporates. The temperature
of the water should be $70 \pm 2.0C$ ($158 \pm 4F$). A nitrogen jet should be
extended into the boiling flask with vacuum and condensing water turned
on. The solvent will evaporate and condense into the receiving flask.

Solvent collected in the receiving flask should be discarded. When most
of the solvent has evaporated, remove the boiling flask from the rotary
evaporator and wash down with 20-25 ml n-pentane. Set the flask aside
until the next pentane wash is required. After evaporation of the third
and final pentane rinse, the recovered material, which contains the
n-pentane solubles and approximately 50 ml of pentane is combined and
used to charge the clay-gel column as described in Section B10.2.

B10.0 FRACTIONATION.

B10.1   Assemble a set of receiving flasks and identify as follows :

    For saturates :

        1 - 500 ml conical flask marked "A".

    For aromatics :

        1 - 500 ml conical flask marked "B".
        1 - 1000 ml conical flask marked "C".

For polar compounds :

    1 - 500 ml conical flask marked "D".
    1 - 1000 ml conical flask marked "E".
    1 - 500 ml conical flask marked "F".

B10.1.1   Prepare the adsorption column (Figure B1) by placing 100g of Attapulgus Clay in the upper section of the column and 200g of silica gel (activated) plus 50g of clay on top of the gel in the lower section.

> Note 5- It is important that the adsorbents in each column be packed uniformly and to a constant level. The upper columnn is packed in two lifts with approximately 50g in each lift. After each lift, pack the column using 12 taps with a soft rubber hammer to the area where the clay is in the column. Continue the tapping a four points approximately 90° apart around the column. The top of the clay is then tapped 20 times to achieve a constant level. After each lift is added to the column, repeat the procedure of turning, tapping the sides, and tapping the top. A rubber hammer may be assembled by fitting a small metal rod, about 260mm long, into the side of a No. 10 rubber stopper. A longer metal rod, about 500mm, can be fitted into the top of a No. 6 rubber stopper for use in tapping the top of the packing material.

B10.1.2   The lower column is packed with approximately three equal lifts of silica gel and one lift of the 50g of clay. Place a piece of glass wool (of about 25 mm loose thickness) over the top surface of the clay in the upper column to prevent agitation and disturbance of the clay while charging the column with eluent solvents. Join the clay over gel columns with a No. 10 rubber stopper.

B10.1.3   Fresh adsorbents should be used for each determination as adsorption capacities become limited after use. The columns should not be packed more than a few hours prior to their use as moisture from the air can be adsorbed on the clay and silica surfaces. In addition, the packed columns should be covered with aluminum foil if the columns remain unused for more than 15 minutes.

156

B10.1.4   Place a preweighed 500 ml conical flask "A" under the column assembly that will be used to collect the saturate fraction. Place another preweighed conical flask "B" to the side so that when the column is disconnected later, the aromatic fraction from the upper column can be collected into that flask.

B10.2   Add 25 ml n-pentane to the top of the clay portion of the assembled column and allow to percolate into the clay. After most of the pentane has entered the column, charge to the column the n-pentane diluted sample that was prepared according to B9.4. See Figure B3.

Note.6- The sample and eluents may be added to the column through a 65 mm diameter long stem funnel (the funnel can remain on top of the column). At no time after the initial charge of pentane has been added should air be allowed to enter the clay bed. This is accomplished by keeping a continuous flow of liquid through the column until the specified volume of effluent is collected. After the columns have been separated, air can be introduced as the subsequent processes involve only the stripping of materials from the adsorbents

Wash the sample flask with n-pentane and add the washings to the column. After most of this material has entered the clay, wash the walls of the column above the clay free of sample with n-pentane.

B10.3   After most of the washings have entered the clay, charge n-pentane to the columns and maintain a head level well above the clay bed to wash the saturate fraction of the sample from the adsorbents. Collect 250 ± 10 ml of the first pentane effluent in flask "A".

B10.4   Disconnect the two column sections, placing the 500 ml flask "B" for collecting the aromatics under the upper or clay column. Place flask "A" under the lower or silica column.

Rinse the walls of the lower or silica gel column and allow to drain for 30 minutes into flask "A", yielding 300 ± 10 ml total effluent. The saturate fraction is in this solution .

Continue washing the upper clay section with n-pentane. Maintain a moderate liquid head level above the clay during this wash and adjust n-pentane additions so that the level is about 25 mm above the clay.

When 250 ml has been collected in flask "B", wash the walls of the column with 10-20 ml n-pentane. Discontinue the n-pentane additions at this point and allow the solvent to drain for 30 minutes. The effluent quantity after draining should be between 275 and 300 ml. The first part of the aromatic fraction is in this solution.

B10.5   Remove the receiver flasks from both columns replacing the lower or silica gel column receiver with a 1000 ml conical flask "C" which will be used to collect additional aromatics.

B10.5.1   The filled receiver flask "A" for the lower column (saturates) should be placed on a hot water bath as described in Section 11.

B10.5.2   The upper clay column receiver should be replaced with another 500 ml flask "D" which will be used to collect the first part of the polar fractions.

Set aside the filled receiver flask "B" for the upper column (aromatics), as the final effluent for aromatics wii be added to this material later.

B10.6   The solvent system for aromatics consists of a acetone-toluene mixture proportioned 20 percent acetone to 80 percent toluene by volume of mixture.

B10.6.1   Pour the acetone-toluene mixture onto the silica column to collect 1000 to 1025 ml effluent.

Replace the filled receiving conical flask "C" with the flask containing the first aromatic effluent (flask "B" that now should contain approximately 300ml of solution)

Charge the lower silica column with the final eluent using approximately 100 ml of pure toluene to insure complete stripping of aromatics from the silica column.

B10.7   The solvent system for polar compounds is an acetone-toluene mixture proportioned 63 percent acetone to 37 percent toluene by volume of mixture.

B10.7.1   Pour the acetone-toluene mixture onto the clay column to collect approximately 500 ml effluent.

Replace the 500 ml polar compound receiver flask "D" with a 1000 ml conical flask "E" and continue to pour polar solvent through the column until approximately 1000 to 1025 ml effluent has been collected.

B10.7.2   Replace this filled receiver "E" with a 500 ml conical flask "F" to receive the final effluent.

Charge the upper clay column with the final eluent using approximately 200 ml of methylene chloride to insure complete stripping of polar compounds from the column.

B11.0   SOLVENT REMOVAL.

B11.1   Solvents are removed from the saturate-solvent solution (flask "A") by the use of a hot water bath (temperature at approximately 71C (160F)).

The receiving flask is adjusted so that the flask is slightly immersed into the water bath. Place a nitrogen jet 40 to 50 mm above the liquid in the flask and open the nitrogen valve to the point that the gas only mildly cuts into the surface of the liquid. Lower the nitrogen jet occassionly during the evaporation but do not place it below the surface of the liquid. When the solvent has been completely evaporated, remove the flask from the water bath.

B11.2   Solvents are initially removed from the aromatic and polar compound solutions by the use of a rotary evaporation system in accordance with Section B9.4. The recovered rotary evaporated material is transferred using two washes of n-pentane (10 to 20 ml each) and a final wash of toluene (10 to 20 ml) to the preweighed conical flask for the appropriate aromatic or polar fraction.

All flasks, including the saturate flask, are then placed on a hot plate at approximately 260C (500F).

Note 7- If a temperature controlled hot plate is not available, a hot plate set on high heat may be used with the flask set on a wire gauze.

Sweep the surface of the residue with nitrogen. Remove the flask from the hot plate when the first trace of smoke appears at the top of the flask.

With saturates and aromatics only one such heating is necessary.
For polars, two heatings will be required.

Place samples in a convection oven at 110C (230F) for 1 1/2 to 2 hours. Flasks should then be placed in a dessicator and cooled to room temperature. After the sample flasks have cooled to room temperature, weigh each flask to determine the amount of residue by subtracting the initial weight of the flask.

B11.3   To determine the asphaltenes residue, weigh the oven-dried Buchner funnel and subtract the initial weight of the funnel, weigh the oven-dried Buchner funnel and subtract the initial weight of the funnel. Weigh the the oven-dried 250 ml conical flask (Section B 9.2) and subtract the initial weight of the flask. Add these two weights to determine the asphaltene residue.

B12.0   CALCULATIONS.

B12.1   Calculate the amounts of asphaltenes (n-pentane insolubles), saturates, aromatics, and polar compounds in the samples as follows ·

Asphaltenes, weight percent $= (B / A) \times 100$

Saturates, weight percent $= (C / A) \times 100$

Aromatics, weight percent $= (D / A) \times 100$

Polar compounds, weight percent $= (E / A) \times 100$

Where :

A = grams of original sample used.

B = grams of sediment filtered from n-pentane-sample mixture (Buchner funnel).

C = grams of residue from n-pentane effluent (flask "A").

D = grams of residue from n-pentane effluent of upper or clay column after column separation plus residue from aromatic solvent effluent of lower or silica gel column residue plus residue from toluene final effluent of lower column (flasks "B" and "C").

E = grams of residue from the 500-1000 ml polar compound effluent plus residue of final effluent of methylene chloride, all from the upper or clay column (flasks "D", "E", and "F").

B12.2   Normalizations.

Normalized percent for asphaltenes = (weight percent of asphaltenes) x F

Normalized percent for saturates = (weight percent of saturates) x F

Normalized percent for aromatics = (weight percent of aromatics) x F

Normalized percent for polar compounds
= (weight percent of polar compounds) x F

Where F = 100 / G, and

G = sum of asphaltene, saturate, aromatic, and polar compound weight percent.

B13.0   PRECISION.

B13.1   There is no precision statement at present. A rule of thumb for a single operator with a single bitumen sample is that the total

calculated percentages of asphaltenes, saturates, aromatics and polar compounds should add to at least 92 percent recovery.

Two runs by the same operator on the same bitumen sample should be suspect if any component is not within one calculated percent of that calculated in the previous run.

B14.0   REPORT.

B14.1   The report should contain calculated and normalized percentages of components. Additionally, the report should contain the approximate amounts of materials collected in each receiver.

B15.0   NOTES.

B15.1   Place approximately 20g of molecular sieves in the reagent containers to absorb water. Insure that sieves are not removed from the reagent container during transfer of the reagent from the container to columns or other test containers.

B15.2   Prior to the day of the test, weigh one 250 ml conical flask, three 500 ml conical flasks and one Buchner funnel. Place the sample in the 250 ml flask and reweigh to obtain sample weight.

B15.3   Prior to the day of the test, place 62.5 ml of n-pentane in the sample flask and allow to stand overnight.

B15.4   Trichloroethylene is used for cleaning of the glassware, especially the fritted disk of the columns and Buchner funnels. The cleaning process is as follows :

> Buchner funnels are cleaned by placing them on filtering flasks and filling the funnel with trichloroethylene twice. Each trichloroethylene wash is followed by three fillings and washings with distilled water allowing the solvent and water to filter through completely before the next trichloroethylene washing.

Columns are washed using two trichloroethylene washes followed by three distilled water washes following the same procedure as for the funnels.

B15.5   In washing the receiver flasks and boiling flasks from the rotary evaporator it is important to use only n-pentane prior to the separation of the columns. After the columns have been split, the washing is conducted with two washings of n-pentane and a final wash of toluene.
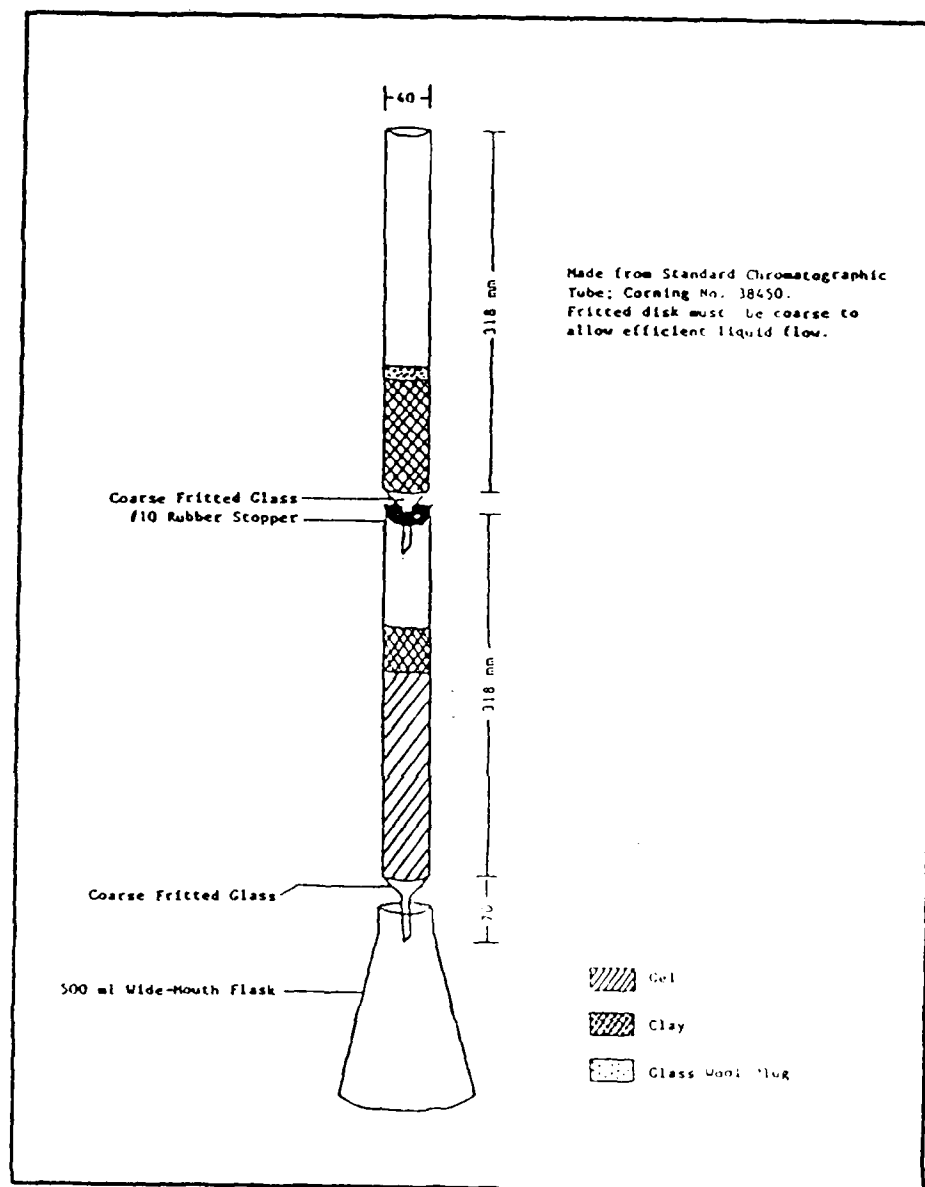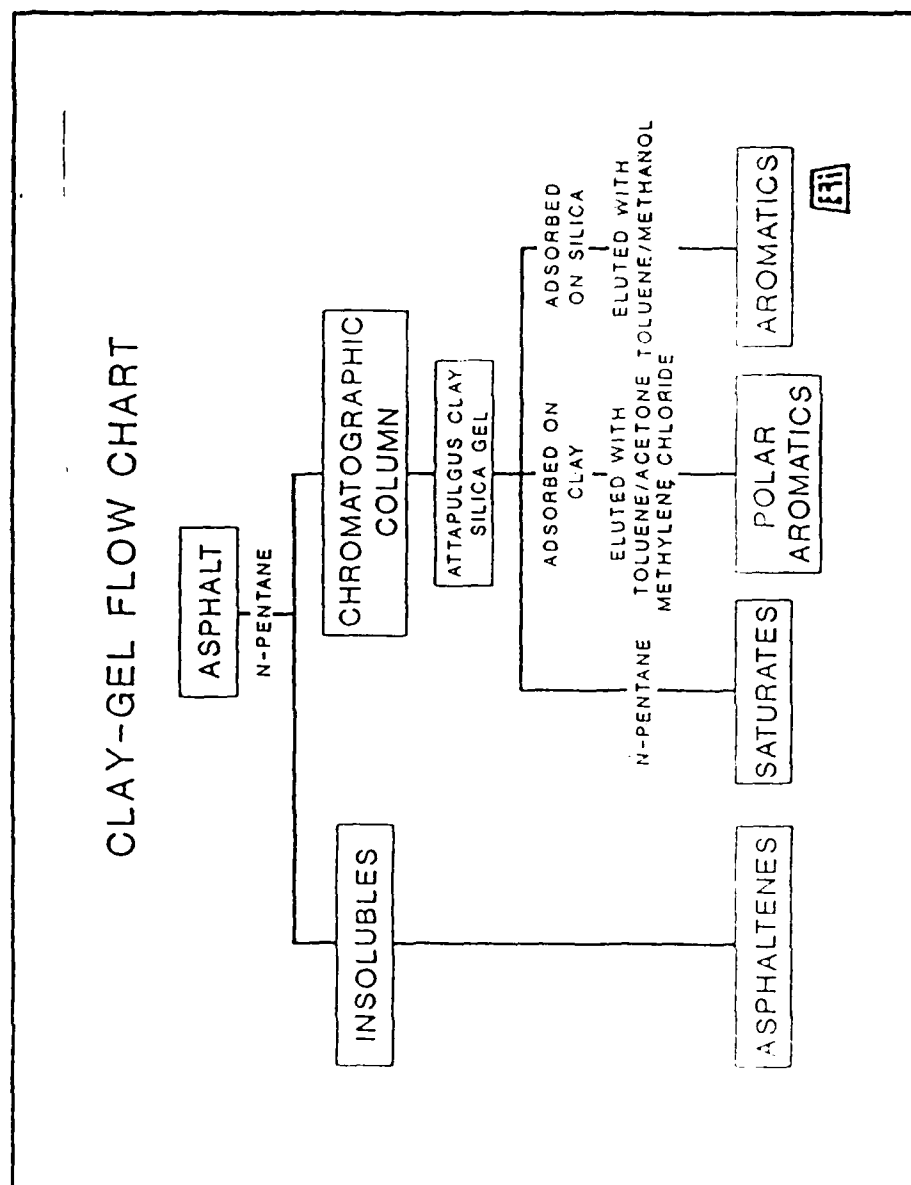
Figure B1. Clay-Gel Apparatus.

CLAY-GEL FLOW CHART

```
                      ASPHALT
                    │ N-PENTANE
          ┌─────────┴─────────┐
     INSOLUBLES        CHROMATOGRAPHIC
                          COLUMN
                    ┌──────────────┐
                    │ ATTAPULGUS CLAY│
                    │   SILICA GEL   │
          ┌─────────┼───────────────┐
          │         │               │
    ADSORBED ON    ADSORBED ON    ADSORBED
      CLAY           CLAY         ON SILICA
                  ELUTED WITH     ELUTED WITH
    N-PENTANE   TOLUENE/ACETONE  TOLUENE/METHANOL
                METHYLENE CHLORIDE
          │         │               │
   ASPHALTENES   SATURATES   POLAR      AROMATICS
                            AROMATICS
```

ASPHALT

N-PENTANE

CHROMATOGRAPHIC COLUMN

ATTAPULGUS CLAY SILICA GEL

ADSORBED ON SILICA

ELUTED WITH TOLUENE/METHANOL

AROMATICS

ADSORBED ON CLAY

ELUTED WITH TOLUENE/ACETONE METHYLENE CHLORIDE

POLAR AROMATICS

N-PENTANE

SATURATES

INSOLUBLES

ASPHALTENES
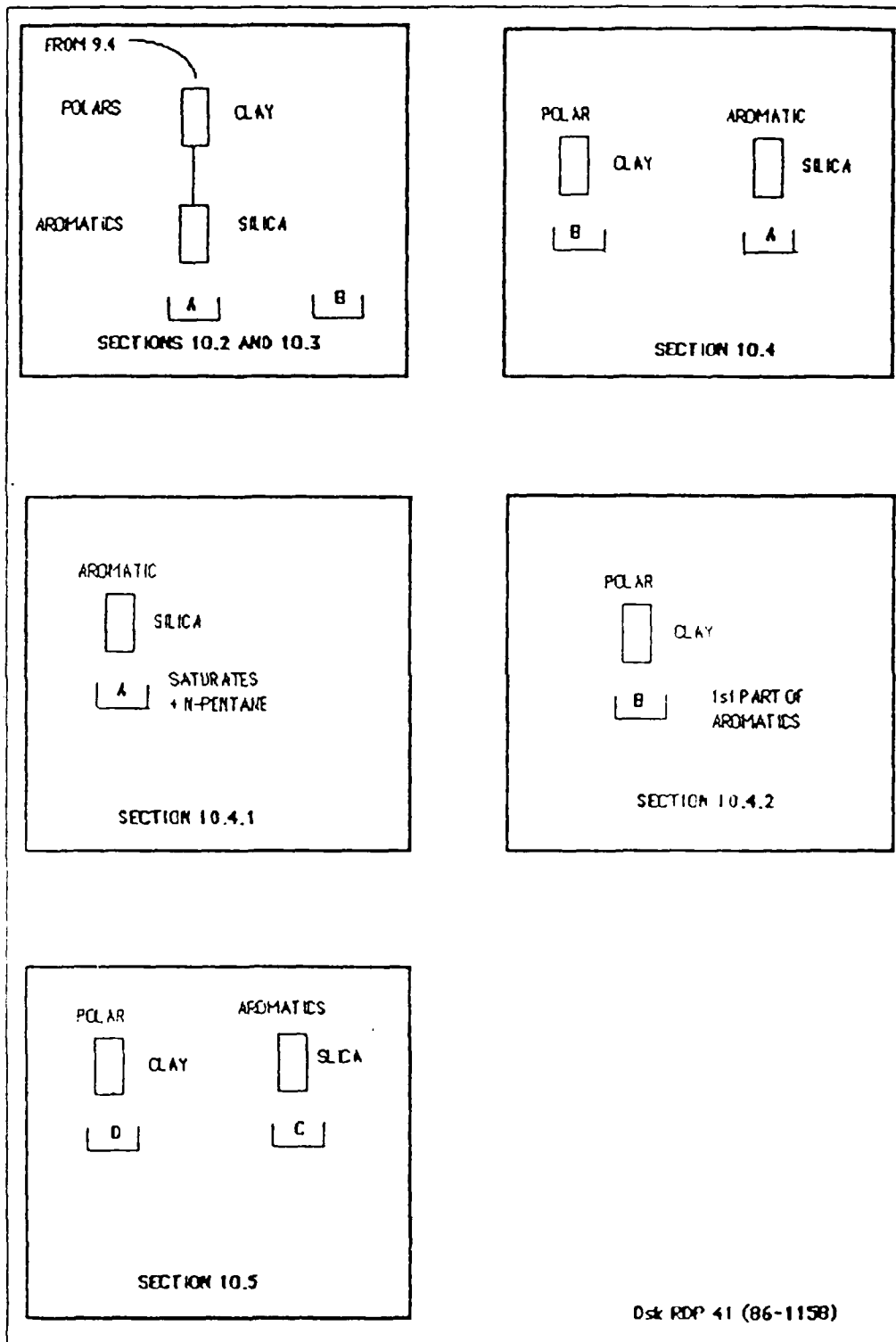
Figure B2. Clay-Gel Flow Chart.

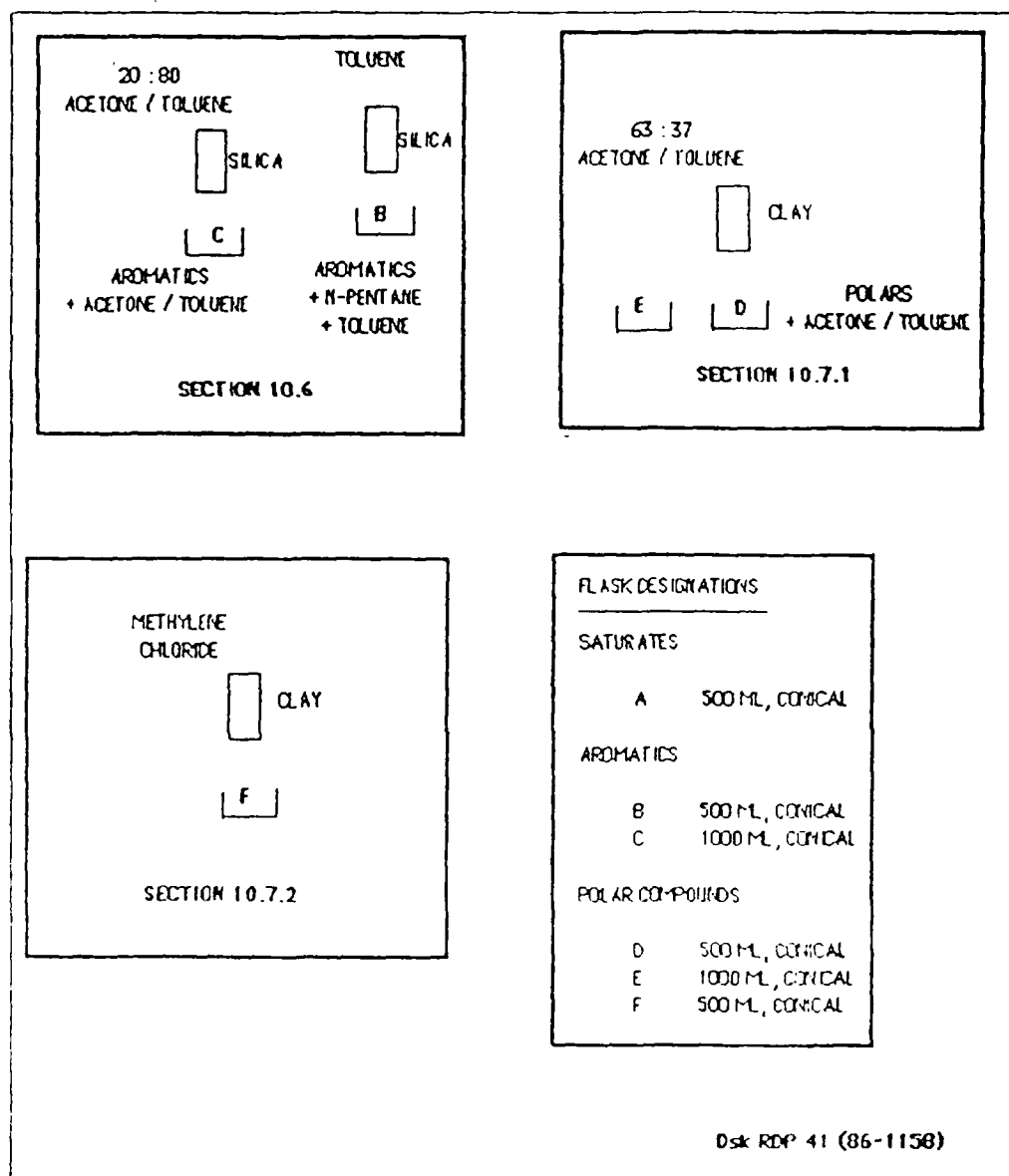Figure B3. Procedure Schematic.

Figure B3. Procedure Schematic (Concluded).

CLAY GEL DATA FORM
ASPHALT CHEMICAL COMPOSITION ANALYSIS ASTM D2002

PROJECT ID _____   DATE _____

SAMPLE ID _____   TECHNICIAN _____

                                        CALC. BY: _____ CHECKED BY: _____

A. **TARE WEIGHTS:**

1. EF # (S) _____   INIT. WT. _____ gm
2. EF # (AR) _____   INIT. WT. _____ gm
3. EF # (PC) _____   INIT. WT. _____ gm
4. BF # (A) _____   INIT. WT. _____ gm
5. EF # (A) _____   INIT. WT. _____ gm

                                      SAMPLE WT. _____ gm

B. **FINAL WEIGHTS:**

1. EF # _____   (S) _____ gm _____ gm
2. EF # _____   (AR) _____ gm _____ cm
3. EF # _____   (PC) _____ gm _____ gm
4. BF # _____   (A) _____ gm _____ gm
5. EF # _____   (A) _____ gm _____ gm

C. **FRACTIONAL COMPOSITION PERCENTAGES**

1. % SATURATES $= \dfrac{\text{wt. (EF + S) - wt. EF}}{\text{Total Sample Weight}} \times 100$

2. % AROMATICS $= \dfrac{\text{wt. (EF + AR) - wt. EF}}{\text{Total Sample Weight}} \times 100$

3. % POLAR CMPDS $= \dfrac{\text{wt. (EF + PC) - wt. EF}}{\text{Total Sample Weight}} \times 100$

4. % ASPHALTENES $= \dfrac{[\text{wt. (BF + A) - wt. BF}] + [\text{wt. (EF + Residue) - wt. EF}]}{\text{Total Sample Weight}}$

                                      TOTAL % _____

NORMALIZATIONS:

	SATURATES	AROMATICS	POLAR CMPDS	ASPHALTENES	TOTAL
NORMALIZED					
AVERAGE					

APPENDIX C

COMPATIBILITY BY HEITHAUS FLOCCULATION

AIR FORCE ENGINEERING AND SERVICES CENTER

TENTATIVE TEST PROCEDURE (AFESC-2)

**DETERMINING COMPATIBILITY OF BITUMINOUS MATERIALS USING
A MODIFIED HEITHAUS FLOCCULATION RATIO TEST PROCEDURE**

Draft of Tentative Test Method (AFESC-2) for

DETERMINING COMPATIBILITY OF BITUMINOUS MATERIALS USING A MODIFIED HEITHAUS FLOCCULATION RATIO TEST PROCEDURE

A1.0   SCOPE.

A1.1 This method covers the measurement of solution properties of bituminous binders by the modified Heithaus Flocculation Ratio Method. This method is for use with recovered aged binders, virgin asphalts, asphaltene-containing modifiers, and blends of recovered aged binders with modifiers and/or virgin binders.

A2.0   APPLICABLE DOCUMENTS.

A2.1   ASTM Standards :

D 70, "Standard Test Method for Specific Gravity of Semi-Solid Bituminous Materials".[2]

D 979, "Standard Methods for Sampling Bituminous Paving Mixtures".[2]

D 1856, "Standard Test Method for Recovery of Asphalt from Solution by Abson Method".[2]

D 2172, "Standard Test Methods for Quantitative Extraction of Bitumen from Bituminous Paving Mixtures".[2]

A3.0   SUMMARY OF METHOD.

A3.1   A 1.0g sample of bitumen or bitumen blend is placed in a 50 ml flask. Four additional 1.0g samples are placed in 125 ml flasks. The sample in the 50 ml flask is solvated in 0.5 ml of reagent grade toluene. The samples in the four 125 ml flasks are solvated in 1.0, 2.0, 4.0, and 6.0ml of the same solvent. To each solution, n-dodecane titrant is added until flocculation is detected.

The volumes of toluene (polar) and n-dodecane (non-polar) recorded for each test are used to determine the following solubility properties :

- Asphaltene Peptizability (P_a).

- Maltene Peptizing Power (P_0).

- State of Peptization of an Asphaltene Dispersion (P).

A4.0   SIGNIFICANCE AND USE.

A4.1   This method provides a means of determining the relative compatibility between blends of bituminous materials. This is particularly crucial in recycling applications which involve blends of field aged binders of unknown origin, modifiers, and/or virgin binders

A5.0   DESCRIPTION OF TERMS.

A5.1   Flocculation ratio (FR) is defined as the minimum volume proportion of non-polar solvent in an polar/non-polar solution necessary to prevent precipitation of asphaltenes [3].

A5.2   Dilution ratio (DR) is the ratio of the total volume of polar and non-polar solvent to the mass of asphalt sample [3].

A5.3   Asphaltene peptizability (P_a) is the ability of the asphaltenes to remain dispersed. This parameter is related to the flocculation ratio and is expressed as $(1 - FR_{max})$ where FR_{max} is the intercept on the FR axis of Figure C1(b).

A5.4 X_{min} is the minimum volume of non-polar solvent (n-dodecane) required to precipitate the least amount of asphaltenes. This parameter can be approximated by T_0 where T_0 is the intercept on the T axis of Figure C2

171

A5.5 Maltene peptizing power (P_0) is the ability of the maltene fraction to disperse the asphaltenes. P_0 is expressed as :
$FR_{max}(X_{min} + 1)$.

A5.6 State of peptization (P) represents the overall solubility properties of the entire solution. This parameter is expressed as : ($P_0 / (1 - P_a)$)) or as ($X_{min} + 1$).

A6.0 APPARATUS.

A6.1 Buret, glass 50 ml with 1/10 subdivisions and teflon bore stopcock.

A6.2 Double buret clamp.

A6.3 Camera with microscope adapter.

A6.4 Dessicator, glass, non-vacuum, 160 mm I.D. x 255 mm and dessicator plate, 140 mm diameter.

A6.5 Filter paper, 7.0 cm, medium.

A6.6 Conical flask, 50 ml and 125 ml.

A6.7 Funnel, filtering, 55 mm top diameter, 63 mm series.

A6.8 Glass rods for making spot tests. 125 mm (5 inch) length.

A6.9 Magnetic stirrers with magnetic stir bars

A6.10 Microscope with transmitted light features and movable stage.

A6.11 Pipets, disposable. For making smears on microscope slides.

A6.12 Ring stands for burets.

A6.13 Thick hanging drop slides, medium, two cavities

A6.14   Cover slides 22 x 22 mm, No. 1 1/2.

A6.15   Timer.

A7.0   REAGENTS AND MATERIALS.

A7.1   N-dodecane, reagent grade.

A7.2   Toluene, reagent grade.

A7.3   Toluene : dodecane mixture, 50 : 50 by weight.

A7.4   Trichloroethylene, technical grade. Used for cleaning glassware.

A8.0   PREPARATION OF SAMPLE.

A8.1   Bitumen for testing should be frozen at 6F (-14C) to facilitate the weighing process. After weighing, bitumen should be warmed in an oven to approximately 110C (230F) for 5 to 10 minutes before introduction of the solvent.

A8.2   Weigh 1.0000 ± 0.0500g sample into a 50 ml flask. Weigh four 1.0000 ± 0.0550g samples into each of four 125 ml flasks.

Dissolve the sample in the 50 ml flask with 0.5 ml of polar solvent (toluene). Dissolve the remaining four samples in 125 ml flasks in volumes of 1.0, 2.0, 4.0, and 6.0 ml of toluene Use a magnetic stirrer to bring samples into solution. Record sample weights and comments using the appropriate data forms. Ambient temperature should be maintained at 70 ± 5F (21 ± 3C).

A9.0   FLOCCULATION OBSERVATIONS.

A9.1   Before beginning titrations, check to determine that all of the sample has gone into solution. If not, continue stirring until all sample is dissolved.

Start with the 1.0, 2.0, 3.0, and 4.0 ml flasks, titrate dodecane in increments of 1.0 ml, see Section A13, "Titration Guide. After titration is completed for these flasks, test the remaining 0.5 ml flask. It is important to record all titrations and comments on the appropriate data sheet.

After each addition of n-dodecane, place flask on magnetic stirrer and stir for 5 minutes. Let mixture set for an additional 5 minutes and observe for flocculate as described below.

A9.2   Set microscope to a range of 100-150x magnification. To prepare microscope slide, dip sample (Note 1) into solution in flask. Smear the tip of the sampler in one of the concave sec.ions of the slide. Care should be taken in smearing the slide. If smear is too thick, light transmission through sample is decreased making microscope viewing difficult. If smear is too thin, then microscope viewing is also difficult. Immediately cover with a cover slip and slide under viewing field of microscope. The observation should be accomplished in less than 30 seconds. Otherwise, extended delay can cause false flocculate results. If there is no flocculate, add the next increment of toluene and repeat the process of A9.1, followed by A9.2.

> Note 1- A sampler can be made by holding a disposable glass pipet over a bunsen burner flame. A small bead will form on the end which is suitable for making the smears on the microscope slides.

A9.3   Flocculate can be described as an agglomerate of particles or clusters that are insoluble. Flocculation occurs as the concentration of non-polar solvent (dodecane) reaches a critical point (the endpoint) where solute particles can no longer remain dissolved in the polar/non-polar solution, thus, these particles begin to precipitate from the solution. In the course of the titration, as the endpoint is approached, particles may appear at the edge of the glass slide. However, a true endpoint is not reached until clusters of floc particles are present throughout the solution. These clusters may be dispersed throughout the solution or create a "carpet-like" appearance in the solution.

The degree of flocculation and the appearance and size of the floc particles may vary from sample to sample. Thus, consistency must be

maintained in defining the endpoint where flocculation occurs. It is critical that the operator be consistent in defining this endpoint.

A9.4 After observing flocculation in the microscope, a spot test should be conducted as follows:

Fill a dessicator with about 1/2 inch of 50 : 50 toluene / n- dodecane by volume mixture but below the level of the porcelain plate.

Note 2- This is to ensure that the dessicator chamber is filled with the same solvent fumes as the test solvents.

Place a 7.0 cm medium filter paper or a Whatman No. 50 filter paper on the porcelain plate. Thoroughly stir the the asphalt solvent mixture with a glass rod and place one drop on each of two places approximately 4 cm apart and immediately cover the dessicator.

If flocculation has occurred on the filter paper, the flocculate will adhere to paper where the spot was placed while the liquid spreads away from the spot. This appears as a dark spot (flocculate) in the center of a lighter brown ring. If flocculation has not occurred, only a relatively uniform light brown spot will develop and no center dark spot will be observed.

This test is used to verify flocculation results when observed with the microscope. If spot test and microscope observations of flocculation do not agree, both tests should be rerun at that increment of n-dodecane with the same combination of bitumen, toluene, and n-dodecane.

A10.0 CALCULATIONS.

A10.1 Calculate S, T, FR,DR, and (1 / DR) for each of the five sample points at flocculation.

$S = A / B$

$T = C / B$

$FR = A / D$

$DR = D / B$

$DR^{-1} = 1 / DR$

Where :

S = ml of polar solvent (toluene) per gram of sample.

A = ml polar solvent.

B = weight of sample in grams.

.T = ml of non-polar solvent (n-dodecane) per gram of sample.

C = ml of non-polar solvent.

FR = flocculation ratio.

D = sum of polar and non-polar solvents.

DR = dilution ratio.

A11.0   REPORT.

A11.1   A plot of flocculation ratio (FR) versus dilution ratio (DR) should yield a curved line as shown in Figure C1(a).

A11.2   A plot of flocculation ratio (FR) versus the inverse of the dilution ratio (DR^{-1}) should yield a straight line as shown in Figure C1(b).

A11.3   PLotting S versus T should result in a straight line as shown in Figure C2.

A11.4   Results are presented in a tabular form showing the following parameters :

P_a = asphaltene peptizability.

P_0 = peptizing power of the maltenes.

P = the state of peptization of an asphaltene dispersion

X_{min} = minimum volume of non-polar solvent required to precipitate asphaltenes.

$T_0 = X_{min}$.

$\cot \theta = T / S$ from Figure C2.

The mathematical relationships between the above parameters are described below :

$$P_a = (1 - FR_{max})$$

$$P_0 = FR_{max} (1 + X_{min})$$

$$P = (1 + X_{min})$$

$$\cot \theta = (T - T_0) / S$$

Where : FR_{max} = ordinate intercept of the relationship FR versus DR^{-1}.

A linear regression of FR versus DR^{-1} should yield an R^2 value greater than 0.95.

A11.5   The report should contain a summary of results on the parameters defined above. Testing personnel should make the plots during or immediately after the procedure. These curves offer the testing personnel a self-check mechanism on the technique.

A12.0   PRECISION.

A12.2   There is no precision statement at the present time. A rule of thumb is that, for the same operator, a variation of 5 percent or less in the same parameter between separate runs on the same material is usually acceptable.

A13.0 TITRATION GUIDE.

A13.1 This is a guide to assist the operator as to when flocculation can be expected for n-dodecane titrations. It is only a guide. For a more accurate estimate, it is advisable to perform a dry run using a 1.0 ml toluene and 1.0g asphalt sample to give a bracket range of the endpoint. The guide is generally a threshold volume below which flocculation has not been observed. Tests should be made using increments that approach the suggested limits.

The 0.5 ml toluene-asphalt sample generally requires longer to solvate than the other samples. Therefore, it is recommended that while the 0.5 ml sample is being stirred with the magnetic stirrer, the operator should proceed to test the 1.0 ml and 2.0 ml toluene-asphalt samples which generally require less than 30 minutes to solvate. After these two samples have been tested, the operator can proceed with the 0.5, 4.0, and 6.0 ml samples.

TOLUENE CONCENTRATION	TITRATION
0.5	This material generally flocculates at about 1 ml less n-dodecane than the 1.0 ml toluene-asphalt sample.
1.0	Add n-dodecane in 1.0 ml increments up to 2.0 ml total. Check for flocculation. If no floc is formed, add 0.5 ml increments of n-dodecane and observe for flocculation until the volume of titrant is 3.0 ml. If no floc is observed, reduce the titrant increments to 0.2 ml and continue
2.0	Approximately 1.0 ml more n-dodecane will be required than was used to produce floc for the 1.0 ml sample
4.0	Approximately 2.0 - 3.0 ml more n-dodecane will be required than was used to produce floc for the 2.0 ml sample.
6.0	Approximately 2.0 - 4.0 ml more n-dodecane will be required than was used to produce floc for the 4.0 ml sample.

Note. If at anytime floc particles appear at the edges of the slide, reduce increments to 0.1 ml additions. Modifiers generally require considerably more titrant to flocculate and asphalts subjected to RTFO and other recovered binders generally require less.

REFERENCES

1. This test procedure is under the jurisdiction of the Engineering and Services Laboratory, Air Force Engineering and Services Center, Tyndall AFB, FL 32403.

2. 1985 Annual Book of ASTM Standards, Volume 4.03.

3. Heithaus, J.J., "Measurement and Significance of Asphaltene Peptization". Symposium on Fundamental Nature of Asphalt. Presented before the Division of Petroleum Chemistry, American Chemical Society, New York, September, 1960.

4. Waxman, M.H., Deeds, C.T., and Closman, P.J., "Thermal Alterations of Asphaltenes in Peace River Tars". Society of Petroleum Engineers of AIME, SPE 9510, September, 1980, pp 1-20. Paper presented at 55th Annual Fall Technical Conference and Exhibition of the Society of Petroleum Engineers of AIME, Dallas, Texas, September 21-24, 1980
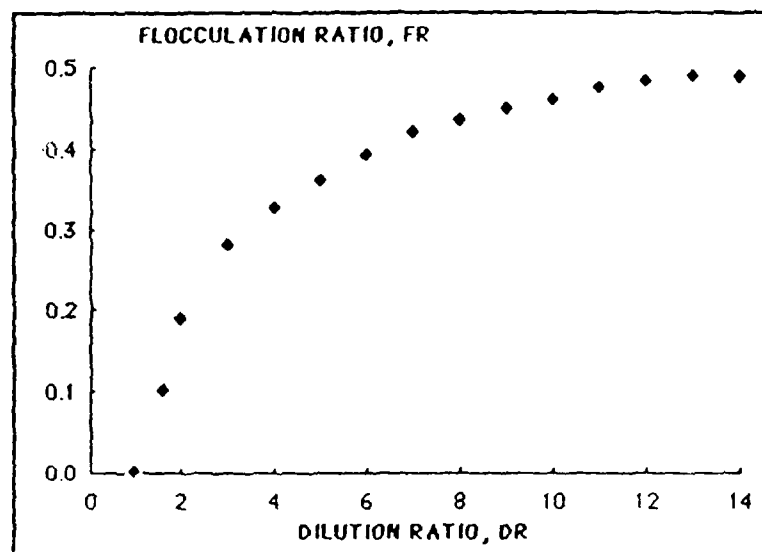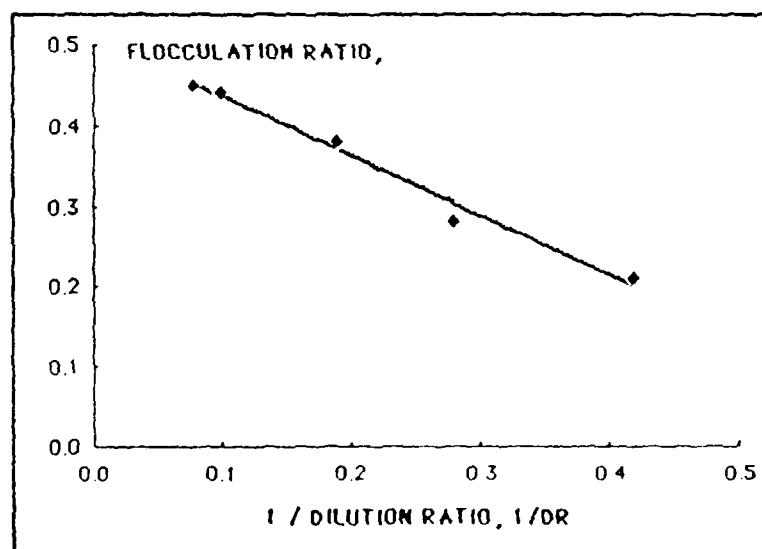
Figure C1(a). Flocculation Ratio versus Dilation Ratio.

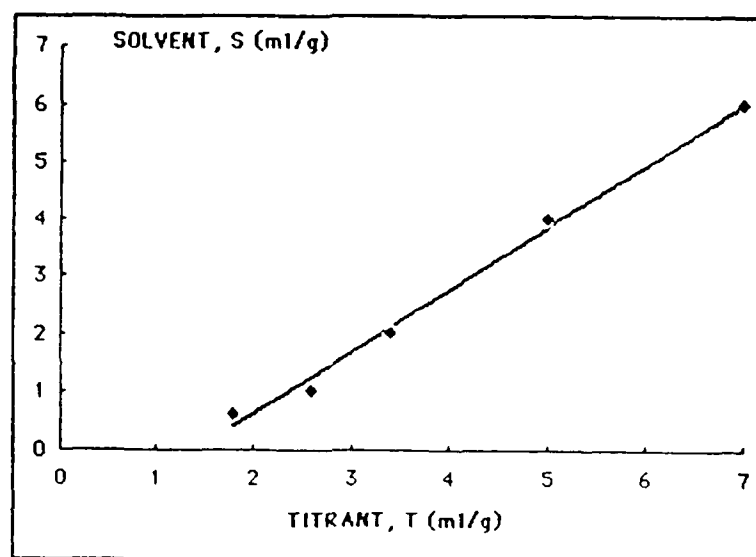Figure C1(b). Flocculation Ratio versus Inverse of Dilution Ratio.

Figure C2. Asphaltene Solubility Test Results.
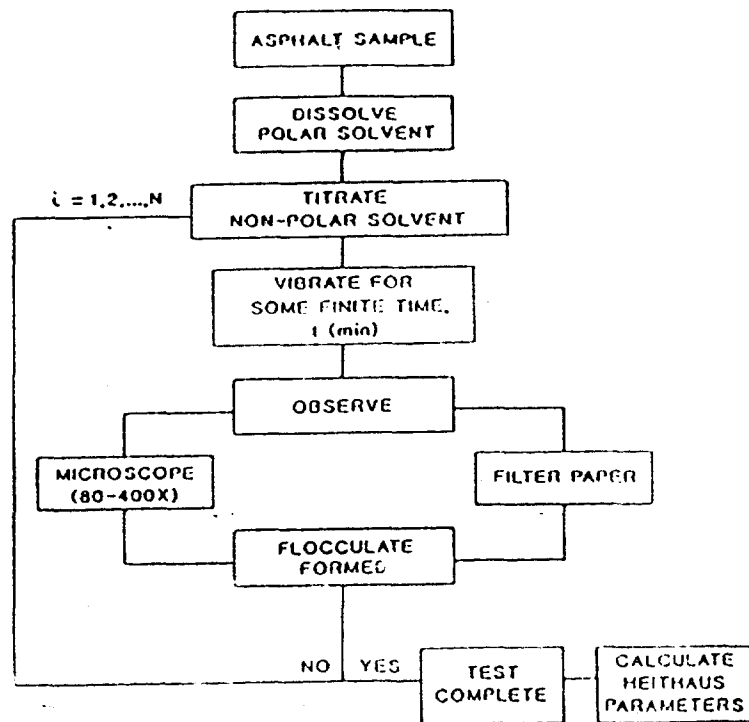
HEITHAUS TEST METHOD
FLOW CHART

```
                    ┌─────────────────┐
                    │  ASPHALT SAMPLE │
                    └────────┬────────┘
                             │
                    ┌────────┴────────┐
                    │    DISSOLVE     │
                    │  POLAR SOLVENT  │
                    └────────┬────────┘
                             │
  i = 1,2,....N     ┌────────┴────────┐
                    │     TITRATE     │
                    │ NON-POLAR SOLVENT│
                    └────────┬────────┘
                             │
                    ┌────────┴────────┐
                    │   VIBRATE FOR   │
                    │ SOME FINITE TIME,│
                    │     t (min)     │
                    └────────┬────────┘
                             │
                    ┌────────┴────────┐
                    │     OBSERVE     │
                    └──┬───────────┬──┘
          ┌────────────┴──┐    ┌───┴──────────┐
          │  MICROSCOPE   │    │ FILTER PAPER │
          │   (80-400X)   │    │              │
          └────────┬──────┘    └──────┬───────┘
                   │    ┌──────────┐  │
                   └────┤FLOCCULATE ├──┘
                        │ FORMED    │
                        └─────┬─────┘
                              │
            NO │ YES   ┌──────┴─────┐  ┌─────────────┐
                       │    TEST    │  │  CALCULATE  │
                       │  COMPLETE  ├──┤   HEITHAUS  │
                       │            │  │  PARAMETERS │
                       └────────────┘  └─────────────┘
```

Figure C3. Heithaus Test Method Flow Chart.

LINEAR REGRESSION

HEITHAUS SOLUBILITY RESULTS	SAMPLE I.D. _____	WAXMAN SOLUBILITY RESULTS
a =		a =
b =		b =
r =		r =
r^2 =		r^2 =

FR v. DR^{-1}		S vs. T	
_____	0.01	_____	1.0
_____	0.1	_____	1.5
_____	0.2	_____	2.0
_____	0.3	_____	2.5
_____	0.4	_____	3.0
_____	0.5	_____	3.5
_____	1.0	_____	4.0
		_____	4.5
		_____	5.0

X Min. = $\left| b/a \right|$ =

P_a = 1 - a =

P = 1 + x min. =

P_o = P · a =

cot θ = $1/b$ =

To = $\left| a/b \right|$ =

CALCULATIONS CHECKED by _____

Note: a = vertical axis intercept

b = slope

HEITHAUS TEST

Date _____   Sample I.D. _____   Polar Solvent _Toluene_

Proj.# _____   N-Polar Solvent _N-Dodecane_

Tsd. by _____   Flask I.D. _____

Weight of Flask + Sample   _____ gm

Weight of Flask   _____ gm

                        Weight of Sample _____ gm

            Volume Polar Solvent: _____ ml

Volume Non-Polar Solvent: _____ ml

BURET READINGS	AMOUNT ADDED	TOTAL	REMARKS

CALCULATIONS:

S = Vol. (Polar Solvent)/Weight of Sample =
T = "   (N-Polar Solvent)/Weight of Sample =
FR = "   (Polar Solvent)/Vol. (Polar + N-Polar Solvents) =
DR = "   ("   + N - Polar Solvents)/Wgt. of Sample =
1/DR =

Calculations Checked by:

APPENDIX D

MODIFIED LOS ANGELES ABRASION

Florida Method of Test
for

RESISTANCE TO ABRASION OF SMALL SIZE COARSE AGGREGATE BY
USE OF THE LOS ANGELES MACHINE

Designation: FM 1-T 096

1. **SCOPE**

   1.1 This method covers a procedure for testing sizes of coarse aggregate smaller than 37.5 mm (1 1/2 in.) for resistance to abrasion using the Los Angeles testing machine.

   1.2 Section 8, Modified Los Angeles Abrasion Test, has been added to AASHTO T 96. This modification shall be used with <u>Lightweight Expanded Aggregate for Bituminous Construction</u>.

   Note 1: A procedure for testing coarse aggregate larger than 19.0 cm (3/4 in.) is covered in the Method of Test for Resistance to Abrasion of Large Size Coarse Aggregate by the Los Angeles Machine (ASTM C 535).

2. **DESCRIPTION OF TERMS**

   2.1 <u>Constant Mass</u> - Test samples dried at a temperature of 110 ± 5 C (230 ± 9 F) to a condition such that it will not lose more than 0.1 percent moisture after 2 h of drying. Such a condition of dryness can be verified by weighing the sample before and after successive 2 h drying periods. In lieu of such a determination, samples may be considered to have reached constant mass when thay have been dried at a temperature of 110 ± 5 C (230 ± 9 F) for an equal or longer period than that previously found adequate for producing the desired constant mass condition under equal or heavier loading conditions of the oven.

3. **APPARATUS**

   3.1 <u>Los Angeles Machine</u> - The Los Angeles abrasion testing machine equipped with a counter and conforming in all its essential characteristics to the design shown in Fig. 1, shall be used. The machine shall consist of a hollow steel cylinder, closed at both ends, having an inside diameter of .711 $\pm$ 5 mm (28 ± 0.2 in.) and an inside length of 508 $\pm$ 5 cm (20 ± 0.2 in.). The cylinder shall be mounted on stub shafts attached to the ends of the cylinder but not entering it, and shall be mounted in such a manner that it may be rotated with the axis in a horizontal position within a tolerance in slope of 1 in 100. An opening in the cylinder shall be provided for the introduction of the test sample. A suitable, dust-tight cover shall be provided for the opening with means for bolting the cover in place. The cover shall be so designed as to maintain the cylindrical contour of the interior surface unless the shelf is so located that the charge will not fall on the cover, or come in contact with it during the test. A removable steel shelf extending the full length of the cylinder

and projecting inward 89 ± 2 mm (3.5 ± 0.1 in.) shall be counted on the interior cylindrical surface of the cylinder, or on the inside surface of the cover, in such a way that a plane centered between the large faces coincides with an axial plane. The shelf shall be of such thickness and so mounted, by bolts or other suitable means, as to be firm and rigid. The position of the shelf shall be such that the distance from the shelf to the opening, measured along the outside circumference of the cylinder in the direction of rotation, shall be not less than 1.27 m (50 in.).

Note 2: The use of a shelf of wear-resistance steel, rectangular in cross-section and mounted independently of the cover, is preferred. However, a shelf consisting of a section of rolled angle, properly mounted on the inside of the cover plate, may be used provided the direction of rotation is such that the charge will be caught on the outside face of the angle. If the shelf becomes distorted from its original shape to such an extent that the requirements given in A1.2 of the Appendix to this method are not met, the shelf shall either be repaired or replaced before additional abrasion tests are made.

3.2 Sieves - Conforming to the Specifications for Wire-Cloth Sieves for Testing Purposes (AASHTO M 92).

3.3 Balance - The balance shall conform to AASHTO M 231, Class E.

3.4 Oven - The oven shall be capable of maintaining a uniform temperature of 110 ± 5 C (230 ± 9 F).

4. ABRASIVE CHARGE

4.1 The abrasive charge shall consist of steel spheres averaging approximately 46.8 mm (1-$27/32$ in.) in diameter and each weighing between 390 and 445 g (Note 3).

4.2 The abrasive charge, depending upon the grading of the test sample as described in Section 4, shall be as follows:

Grading	Number of Spheres	Mass of Change, g
A	12	5000 $\pm$ 25
B	11	4584 $\pm$ 25
C	8	3330 $\pm$ 20
D	6	2500 $\pm$ 15

Note 3: Steel ball bearing 46.0 mm (1-$13/16$ in.) and 47.6 mm (1-$7/8$ in.) diameter, weighing approximately 400 to 440 g each, respectively, are readily available. Steel spheres 46.8 mm (1-$27/32$ in.) in diameter weighing approximately 420 g may also be obtainable. The abrasive charge may consist of a mixture of these sizes conforming to the weight tolerances of Paragraphs 4.1 and 4.2.

5. TEST SAMPLE

5.1 The test sample shall consist of clean aggregate representative of the material under test. If the aggregate is dirty or coated, wash until clean (Note 5). The aggregate shall be dried to constant mass (see Section 2), separated into individual size fractions, and recombined to the grading of Table 1 most nearly corresponding the the range of sizes in the aggregate as furnished for the work. The mass of the sample prior to test shall be recorded to the nearest 5 g.

6. PROCEDURE

6.1 The test sample and the abrasive charge shall be placed in the Los Angeles abrasion testing machine and the machine rotated at a speed of 30 to 33 rpm for 500 ± 1 revolutions. The machine shall be so driven and so counter-balanced as to maintain a substantially uniform peripheral speed (Note 4). After the prescribed number of revolutions, the material shall be discharged from the machine, and a preliminary separation of the sample made on a 4.75 mm sieve. The finer portion shall then be sieved on a 1.70 mm (No. 12) sieve in a manner conforming to Section 5.1 of AASHTO T 27, Method of Test for Sieve Analysis of Fine and Coarse Aggregates. The material coarser than the 1.70 mm sieve shall be washed, dried to a constant mass (see Section 2), and weighed to the nearest 5 g (Note 6).

Note 4: Back-lash or slip in the driving mechanism is very likely to furnish test results which are not duplicated by other Los Angeles abrasion machines producing constant peripheral speed.

Note 5: If the aggregate is essentially free from adherent coatings and dust, the requirement for washing before and after test may be waived. Elimination of washing after test will seldom reduce the measured loss by more than about 0.2 percent of the original sample mass.

Note 6: Valuable information concerning the uniformity of the sample under test may be obtained by determining the loss after 100 revolutions. This loss should be determined without washing the material coarser than the 1.70 mm sieve. The ratio of the wash after 100 revolutions to the loss after 500 revoluations should not greatly exceed 0.20 for material of uniform hardness. When this determination is made, care should be taken to avoid losing any part of the sample; the entire sample, including the dust of abrasion, shall be returned to the testing machine for the final 400 revoluations required to complete the test.

7. CALCULATION

7.1 The difference between the original mass and the final mass of the test sample shall be expressed as a percentage of the original mass of the test sample. This value shall be reported as the percentage of wear (Note 7).

Note 7: The percentage of wear determined by this method has no known consistent relationship to the percentage of wear for the same material when tested by ASTM C 535.

187

TABLE D1. GRADING OF TEST SAMPLES

Sieve Size		Mass of Indicated Sizes, g Grading			
Passing	Retained on	A	B	C	D
mm					
37.5 (1-1/2 in.)	25.0 (1 in.)	1 250 + 25	---	---	---
25.0 (1 in.)	19.0 (3/4 in.)	1 250 + 25	---	---	---
19.0 (3/4 in.)	12.5 (1/2 in.)	1 250 + 10	2 500 + 10	---	---
12.5 (1/2 in.)	9.5 (3/8 in.)	1 250 + 10	2 500 + 10	---	---
9.5 (3/8 in.)	6.3 (1/4 in.)	---	---	2 500 + 10	---
6.3 (1/4 in.)	4.75 (No. 4)	---	---	2 500 + 10	---
4.75 (No. 4)	2.36 (No. 8)	---	---	---	5 000 + 10
Total -----------------		5 000 + 10	5 000 + 10	5 000 + 10	5 000 + 10
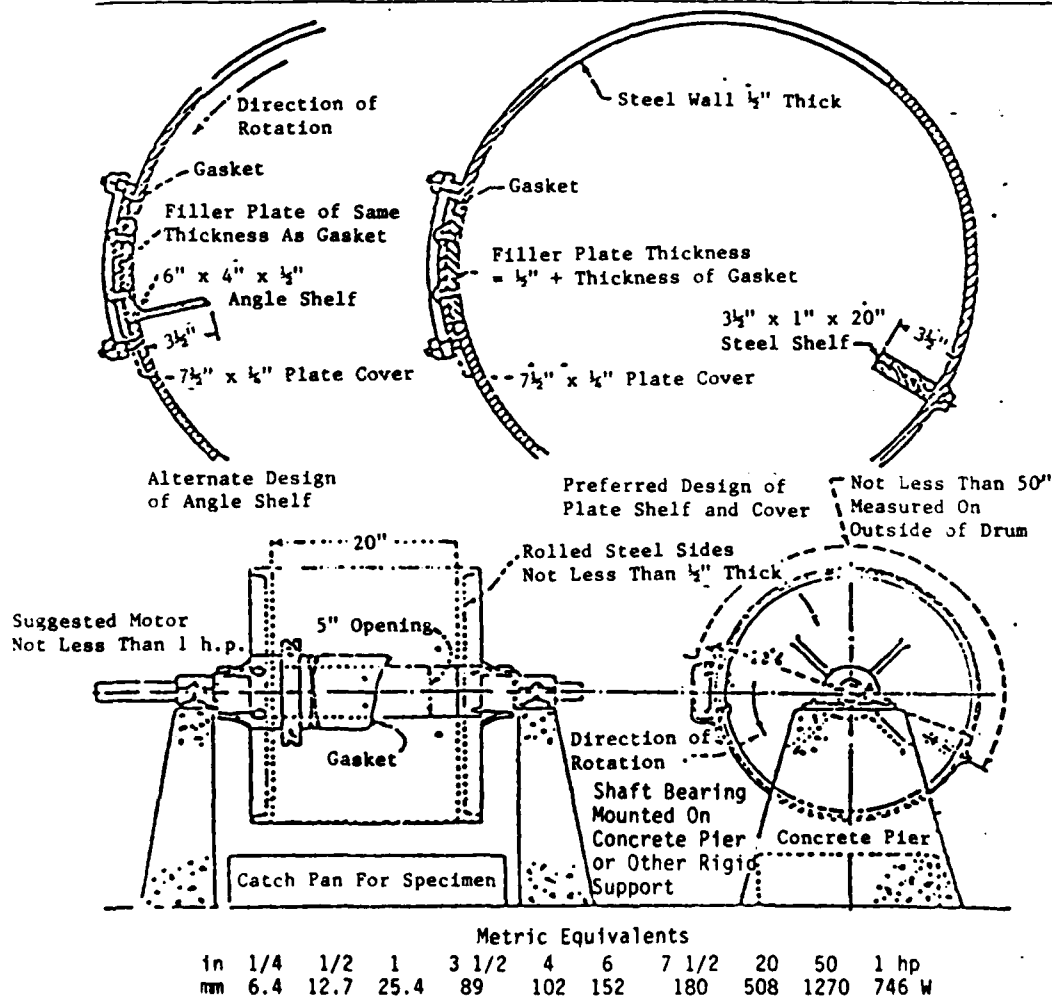
Figure D1. Los Angeles Abrasion Testing Machine.

Metric Equivalents

in	1/4	1/2	1	3 1/2	4	6	7 1/2	20	50	1 hp
mm	6.4	12.7	25.4	89	102	152	180	508	1270	746 W

188

8. MODIFIED LOS ANGELES ABRASION TEST

8.1 This modification shall be used with Lightweight Expanded Aggregate f Bituminous Construction.

8.2 Determine the dry-loose unit weight (U_L) of the lightweight aggregate

8.3 Assume the average unit weight of conventional aggregate to be 90 pcf

8.4 Reduce the lightweight aggregate sample by the following formula:

$$\frac{U_L}{90} = \frac{X}{C}$$

Where: U_L = loose unit weight of lightweight aggregate in pcf,

C = weight of conventional aggregate required for grading in standard test methods,

X = reduced lightweight aggregate sample charge.

8.5 Reduce the abrasive charge by the following formula:

$$\frac{U_L}{90} = \frac{X_1}{C_1}$$

Where: U_L = loose unit weight of lightweight aggregate in pcf,

C_1 = weight of abrasive charge required for grading in standard test methods,

X_1 = reduced abrasive charge for lightweight aggregate.

8.6 Proceed according to standard test method.

Note 8: This modification is necessary for lightweight aggregate to avoid excessive volume of material in the testing machine.

Note 9: Since it is usually impossible to obtain the exact abrasive charge with the steel balls available, obtain the closest abrasive charge possible to the reduced value and then adjust the weight of the sample in proportion to the new abrasive charge.

189

ATTACHMENT

A1. MAINTAINENCE OF SHELF

A1.1 The shelf of the Los Angeles Machine is subject to severe surface wear
and impact. With use, the working surface of the shelf is peened by the
balls and tends to develop a ridge of metal parallel to and about 32 mm
(1-1/4 in.) from the junction of the shelf and the inner surface of the
cylinder. If the shelf is made from a section of rolled angle, not only
may this ridge develop but the shelf itself may be bent longitudinal or
transversely from its proper position.

A1.2 The shelf should be inspected periodically to determine that it is not
bent either lengthwise or from its normal radial position with respect to
the cylinder. If either condition is found, the shelf should be repaired
or replaced before further abrasion tests are made. The influence on the
test result of the ridge developed by peening of the working face of the
shelf is not known. However, for uniform test conditions, it is recommen-
ded that the ridge be ground off if its height exceeds 2 mm (0.1 in.).

190

Tentative Addition to FM 1-T 096,
RESISTANCE TO ABRASION OF SMALL SIZE COARSE AGGREGATE
BY USE OF THE LOS ANGELES MACHINE

* * * * * * * * * * * * * *

1. SCOPE

    1.3 Section 10, Modified Los Angeles Abrasion Test - Fine Aggregate has also
         been added to FM 1-T 096.

10. MODIFIED LOS ANGELES ABRASION TEST - Fine Aggregate

    10.1 Abrasive Charge - Six steel spheres as for grading D and conforming to 4.
          shall be used.

    10.2 Test Sample - The test sample shall be prepared as in 5.1 except that
          Table II below shall be used instead of Table I. Additionally, in 5.1,
          delete the second sentence including the reference to Note 5.
          Sample is to be washed.

TABLE D2.   GRADING OF TEST SAMPLES--FINE AGGREGATE

Sieve Size		Mass of Indicated Sizes, g Grading	
Passing	Retained On	J	K
mm			
4.75 (No. 4)	1.18 (No. 16)	5000 ± 10	---
1.18 (No. 16)	0.600 (No. 30)	--------	5000 ± 10

    10.3 Procedure - Procedure is the same as 6.1 except that a 0.300 mm (No. 50)
          sieve shall be used instead of a 1.70 mm (No. 12) sieve for sieving.

    10.4 Calculation - Determine the percentage of wear as in 7.1.

191

(The reverse of this page is blank.)